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Methodology for Analyzing Crack Initiation in a Vessel under LBE Environment

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Master in Engineering and Quality Management

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DECLARATION

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ABSTRACT

The Belgian Nuclear Research Centre, SCK•CEN, was founded in 1952. This gave the academic world and Belgian industry access to the worldwide development of nuclear energy. Ever since SCK•CEN has been developing a pioneering role with unique achievements and groundbreaking work. The Centre has been developing activities related to MYRRHA, a Multi-purpose Hybrid Research Reactor for High-tech Applications able to work both in subcritical (ADS) as in critical modes. This reactor can contribute in areas as health care when producing radioisotopes for nuclear medicine.

One of the main problems identified by researchers about the reactor is the crack initiation in the vessel in a Lead-bismuth Eutectic (LBE) environment, fact that in long-term affects directly the proper function of the reactor. Because of its relevance, the crack initiation needs reactor MYRRHA to be studied. Keeping that as a principal objective, this dissertation reviews the concept of cracks initiation in terms of lifecycle and analyzes the different project on the topic to classify the possible factors influencing this crack initiation.

The concept of experimental design appears as an alternative to adequately guide future studies on crack initiation and with which reliable results can be provided, in addition to it, the accelerated testing is also proposed as the technique to use for reducing the experiment development time.

An experimental design was developed and a sample size was defined, estimated through the factors selected as relevant and considering one repetition, but the possibility of increasing or reducing the number of factors besides the levels of the same is left open.

KEYWORDS

Reliability, Accelerated testing, experimental design, Crack initiation

RESUMO

O Centro Belga de Pesquisa Nuclear, SCK • CEN foi fundado em 1952. Isto deu ao mundo académico e industrial Belga acesso ao desenvolvimento mundial da energia nuclear. Desde então o SCK • CEN tem desempenhado um papel pioneiro com realizações únicas e trabalho innovador. O centro tem desenvolvido atividade relativamente ao MYRRHA, Reator de Pesquisa Híbrido Multiusos para Aplicações de Alta Tecnologia, capaz de trabalhar tanto em subcritico (ADS) como em modos críticos. Este reator pode contribuir em áreas como cuidados de saúde para a produção de radioisótopos.

Um dos principais problemas identificados pela investigação realizada acerca do reator é a iniciação de fissuras no ambiente LBE, facto que, a longo prazo, afeta diretamente a função adequada do reator. Por sua relevância, o reator MYRRHA precisa ser estudado. Mantendo isso como um dos principais objetivos, esta dissertação analisa o aparecimento de fissuras na cuba do reator nuclear. O conceito de projeto experimental aparece como uma alternativa para orientar adequadamente estudos futuros sobre iniciação de crack e com o qual resultados confiáveis podem ser obtidos, além disso, os testes acelerados também são propostos como a técnica a ser usada para reduzir o tempo de desenvolvimento da experiência.

Foi definido um plano experimental e proposto um tamanho de amostra que considera uma repetição de cada teste, estimado através dos fatores selecionados como relevantes, mas fica em aberto a possibilidade de aumentar ou reduzir o número de fatores além dos níveis do mesmo.

PALAVRAS-CHAVE:

Fiabilidade, Accelerated testing, experimental design, Iniciação de fissuras.

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List of Abbreviations and Acronyms

ABTs	Accelerated Binary Tests
ABWR	Advanced boiling water reactor
AD	Accelerator Driven
ADDTs	Accelerated Destructive Degradation Tests
ADS	Flexible experimental accelerator Driven System
AGREE	Reliability of Electronic Equipment
ARMDTs	Accelerated Repeated Measures Degradation Tests
ASME	American Society of Mechanical Engineers
AT	Accelerated test
BWR	Boiling water reactor
CANDU	Canadian Deuterium Uranium
DO	Dissolved oxygen
DOE	Design of Experiments
DSA	Dynamic strain ageing
EPR	European power reactor
EST	Environmental stress testing
HALT	Highly accelerated life tests
LBE	Lead-alloy coolants
LME	Liquid metal embrittlement
LMFRs	Liquid metal cooled fast reactors
LWR	Light water reactor
PVC	Polyvinyl chloride
PHWR	Pressurized heavy water reactor
PWR	Pressurized water reactor
S	Stress
SCK•CEN	Belgian Nuclear Research Centre
STRIFE	Stress life
QualAT	Qualitative accelerated tests
QuanAT	Quantitative accelerated tests
UV	Ultraviolet

1. INTRODUCTION

1.1 Scope

Multi-purpose hYbrid Research Reactor for High-tech Applications (MYRRHA) is the flexible experimental Accelerator Driven System (ADS) in development at Belgian Nuclear Research Centre, SCK•CEN. There are many ways to classify a reactor and one of them is through the type of coolant. For MYRRHA, Lead Bismuth Eutectic (LBE) that has been used as primary coolant in Soviet sub-marine reactors, LBE or related lead-alloy coolants could provide significant advantages in terms of safety and economy of Generation IV reactors and accelerator driven systems, thanks to high boiling temperature, chemical inertness with respect to water and potential for natural convection (Baeten, Schyns, Fernandez, De Bruyn, & Van den Eynde, 2014).

A literature review on MYRRHA with LBE environment and crack initiations, in or outside SCK•CEN, shows a huge number of papers that address this topic, through basic statistical analysis. One of the most relevant papers was developed by the Argonne National Laboratory which addresses the fatigue crack initiation in light water reactor (LWR) environment. In this article specific factors are evaluated and statistics are used to process the information. In SCK•CEN, researches have also been developed, one of them was performed by Pierre Marmy and Xing Gong(2012), that addressed the influence of LBE on the fatigue properties of 316L steel. In the case of this paper, descriptive statistic are used to analyze and process the data.

The literature about this area presents different researches that lack the use of inferential statistics (such as hypothesis tests, regression, Bayes) and of a proper sample collection planning. However, it is not possible to have credible results in a regression model if the internal validity is nonexistent in the study, which means it has unknown errors and high uncertainty. So, sample planning is required to extract more information from the data collected, and so it is possible to reduce the repetition of the same experiment. To obtain credible results that consequently could be used to analyze a population (Slack & Draugalis, 2001).

A proposal to increase the inter validity can be the use of experimental design with accelerate testing. Experimental design is a complete investigation tool in which combinations of the levels of factors are investigated.

On the other hand, the use of accelerated life testing reduces the test time significantly while the use of factorial design, by testing the effect of several factors simultaneously, provides the maximum possible information from a minimum number of tests.

1.2 Objectives

The objective of the dissertation is to propose a methodology as an alternative to the common techniques used for the analysis of the most important factors that influence the crack initiation under LBE environment. The specific objectives to achieve are:

- ✓ Perform a theoretical review about SCK•CEN researches and the main experiments for this project.
- ✓ Propose statistical analysis techniques for the data obtained in SCK•CEN.
- ✓ Determine the most important factors to be analyzed for the study of the crack initiation in LBE environment.
- ✓ Propose a technique that allows to plan and later to analyze experiment for the study of cracks initiation in LBE.

1.3 Methodology

For the proper fulfillment of the objectives the next steps were developed:

Phase 1. Literature review - In the development of a research, the literature review is important because it allows the introduction of the reader to the general concepts that are going to be used in the investigation. Additionally for this project It played an important role since it allowed to know according to other projects the most relevant factors and thus to take decisions in the future. For that reason, in this investigation the literature review was performed minutely through searching in journals, books, thesis, proceedings, technical reports and websites.

Phase 2. Review SCK•CEN researches - Data analysis and identification of statistical methodology. In this phase are evaluated some projects through statistical analysis identified the problems of using the techniques after not applying a proper sample size analysis and the Identification of the relevant factors in crack initiation. There is a huge quantity of factors that could have influence in the crack initiation, but those are not standardly clear. A statistical analysis can give the tools for reducing the number of factors in a future experiment for the crack initiation.

Phase 3. In this phase a methodology to analyze and process the data is approached. Correct technique allows to control the deficiency of statistical techniques. Therefore, a series of analysis was made to data obtained and analyzed in previous projects. And with it leave a research base that can be used as a tool in the future. Finalizing with the conclusions.

1.4 Dissertation structure

For the collection and subsequent analysis of data that allows the analysis of the number of cycles until failure occurrence, in this case crack initiation, this dissertation has 6 chapters.

Chapter I presents first an introduction of the project, with a theoretical framework. After, the description of the objectives, the methodology used in this project and the dissertation structure are presented.

Chapter II addresses the nuclear environment concepts, in addition to the introduction of concept and tools such as: reliability, experimental design and accelerating testing.

Chapter III presents SCK•CEN which is the organization where the research was performed. Then an introduction to MYRRHA is given. And later, two projects from SCK•CEN are discussed.

Chapter IV is about statistical analysis of two databases that were previously processed in SCK•CEN.in order to see application examples in data rates to this project, make conclusions.

Chapter V begins with the statement of the problem to be analyzed. Then, the independent and dependent factors are identified, as well as the levels and ranges of the factors. At least, the experimental design is approached.

Chapter VI presents the conclusions of the dissertation.

2. LITERATURE REVIEW

This chapter allows to give a critical vision about the important topics for this project and the theoretical concepts about Nuclear (comprehension of Nuclear, type of reactor, fatigue) and Reliability.

2.1 Nuclear reactor

A nuclear reactor is a device, designed to produce and sustain a long term controlled fission chain reaction, made with carefully selected and strategically placed collection of various materials. The classification of reactors varies and are generally based on the following: type of fission reaction (thermal, epithermal, and fast reactors), purpose of the reactor (power reactors, research reactors, and test reactors), type of the coolant present (such as light/heavy water reactors, gas-cooled reactors, and liquid metal-cooled reactors), type of core construction (cubical, cylindrical, octagonal, and spherical reactors), and so forth. Nuclear reactors have been built for the primary purpose of electricity production, although they are used for desalination and radioisotope production (Charit & Murty, 2013).

Examples of nuclear reactor are presented in Figure 1.

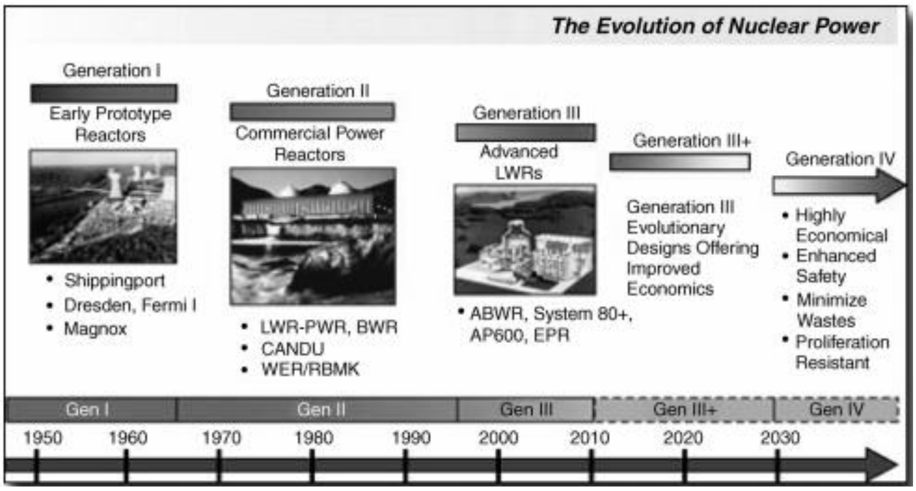

Figure 1: Evolution of nuclear Power

- ✓ Generation-I reactors were built in the initial period of nuclear power expansion and generally had primitive design features. Most of these reactors have either been shut down or will be soon done so. Examples of such reactors are Magnox reactor (Calder Hall reactor in the United Kingdom) and first commercial power reactor at Shippingport in

1957 (in the state of Pennsylvania in the United States). As an example, the Magnox Reactor is a notable Generation-I gas-cooled reactor. Early breed of this reactor was used for plutonium production (for atomic weapons) as well as electricity generation.

- ✓ Generation-II reactors are in most of the commercial nuclear power plants operating today. There are also the reactors employed in naval vessels (such as aircraft carriers and submarines) and many research/test reactors are of this type. The Generation-II reactors incorporated improved design and safety features and productivity over Generation-I reactors. In the Western Hemisphere, a majority of commercial nuclear power plants has Light water reactor (LWR), both pressurized water reactor (PWR) and boiling water reactor (BWR). It is important to remember that LWRs were also built as Generation-I reactors (such as Shippingport facility with 60 MWe power capacity), however most of them are no longer in operation. Another variety is the CANDU (Canadian Deuterium Uranium) reactor, which is basically a pressurized heavy water reactor (PHWR). There are a few different versions of pressurized water reactors (e.g., RBMK type) in Russia and former Soviet-bloc countries.
- ✓ Generation-III and III+ Reactors are being built or will be built within a few years. These are mainly advanced LWRs. Examples include advanced boiling water reactor (ABWR) and evolutionary or European power reactor (EPR). In the same line, Generation III+ category aims to provide reactor systems that have much improved designs and safety features, and much greater capacities.
- ✓ Generation-IV reactors are the futuristic reactors for which research and development efforts are currently in progress. These reactors will be more efficient, safer, longer lasting (60 years and beyond), proliferation-resistant, and economically viable compared to the present nuclear reactors (Charit & Murty, 2013).

2.2 Coolant

The choice of the coolant is one of the main technical issues concerning fast reactors design, since it determines design approach as well as technical and economic characteristics of the system. Moreover, several aspects concerning coolants are related to the reliability and safe operation of liquid metal cooled fast reactors (LMFRs). These aspects are in the field of coolant quality control, materials compatibility, thermal-hydraulics behavior in different operation regimes, and innovative and robust instrumentation and measurement techniques development.

The coolant quality control is of primary importance since it has a direct impact on the structural materials compatibility performance, on the thermal-hydraulics performance of the core, the primary system and the heat exchanger and, to some extent, on maintenance and decommissioning procedures. The study of the physical and chemical properties of the liquid metal and the evaluation of contamination sources which can occur in the reactor system are essential data for the development of technologies to control the coolant quality (IAEA, n.d.).

2.3 Fatigue

Fatigue, as understood by materials technologists, is a process in which damage accumulates due to the repetitive application of loads that may be well below the yield point. The process is dangerous because a single application of the load would not produce any ill effects, and a conventional stress analysis might lead to an assumption of safety that does not exist.

In one popular view of fatigue in metals, the fatigue process is thought to begin at an internal or surface flaw where the stresses are concentrated, and consists initially of shear flow along slip planes. Over several cycles, this slip generates intrusions and extrusions that begin to resemble a crack. A true crack running inward from an intrusion region may propagate initially along one of the original slip planes, but eventually turns to propagate transversely to the principal normal stress.

The modern study of fatigue is generally dated from the work of A. Wöhler, a technologist in the German railroad system in the mid-nineteenth century. Wohler was concerned by the failure of axles after various times in service, at loads considerably less than expected. A railcar axle is essentially a round beam in four-point bending, which produces a compressive stress along the top surface and a tensile stress along the bottom (see Figure 2). After the axle has rotated a half turn, the bottom becomes the top and vice versa, so the stress on a particular region of material at the surface varies sinusoidally from tension to compression and back again. This is now known as fully reversed fatigue loading (Roylance, 2001).

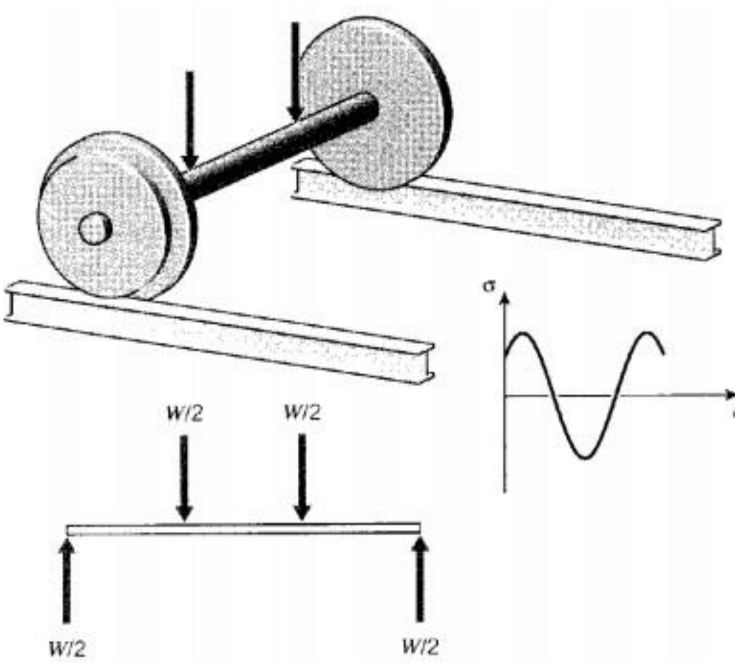

Figure 2: Stress description (Roylance, 2001).

2.3.1 ε-N curve

When local plastic deformations are present during the fatigue process, for example at stress raisers (edges, discontinuities, etc.) the so called strain-based must be applied as an alternative of the stress based approach because the first one does a better characterization of the fatigue, particularly in the low cycle fatigue. The strain-based approach demands more material related information, including the ε-N curves, which represent the mean lifetime as a function of the strain range for a constant stress or strain level, N represents the number of cycles (see Figure 3).

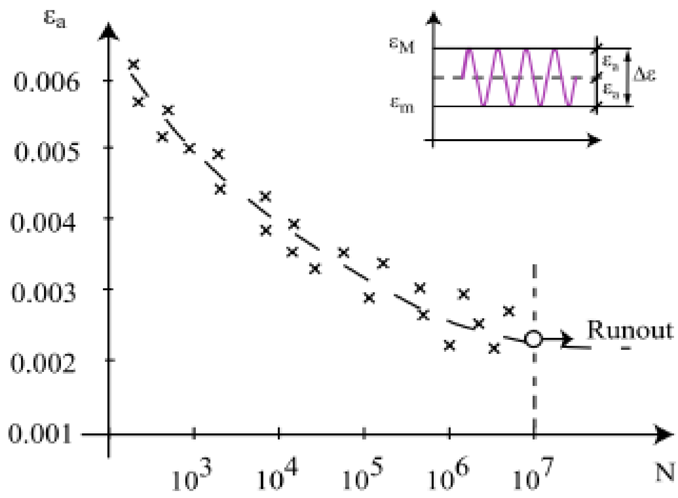

Figure 3: Fatigue for constant strain rate (Castillo & Fernández-Canteli, 2009)

A typical example of fatigue data for constant strain range is provided in Figure 3, and estimates of the ε-N mean and some percentile curves have been added in Figure 4. A detailed analysis of these figures shows the following facts:

1. Fatigue lifetime increases with decreasing strain range;
2. The data exhibit all over the lifetime range a positive curvature (concave from above);
3. Data also suggest that the scatter of fatigue lifetime increases with decreasing strain range;
4. Below a certain strain range, no fatigue failure is expected;
5. The fatigue lifetime has a random character. Thus, instead of a single ε-N curve (the mean) it seems better to use a family of ε-N curves, associated with the corresponding percentiles. So, for simplicity's sake we will refer to the associated ε-N curves (Castillo & Fernández-Canteli, 2009).

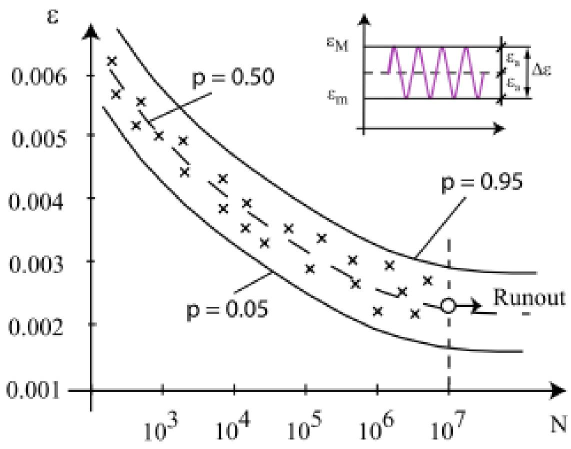

Figure 4: ε-N mean Curve (Castillo & Ferna´ndez-Canteli, 2009)

2.3.2 Fatigue ε-N Data with different environments

The environment, in this case the type of coolant used for the reactor, has changed over time. Air and light water are important coolants that have been studied and these studies can be a base for the study of LBE. The characteristic of the fatigue for those environments are presented below.

- ✓ Air environment

A comparison between statistical models performed by the American Society of Mechanical Engineers ASME, showed that the fatigue ε-N data indicate that fatigue life of austenitic strainless steels in air is independent of temperature in the range from room temperature to 427°C. Although the effect of strain rate on fatigue life seems to be significant at temperature above 400 °C variation, in strain rate in the range of 0.4 - $0.008s^{-1}$ has no effect on the fatigue lives of strainless steels at temperature up to 400 °C.

During cycling loading of the testing, austenitic strainless steels exhibit hardening during the first 50-100 cycles, the extent of hardening increases with increasing strain amplitude. A decreasing temperature and strain rate showed that for the two types of strainless steels cyclic hardening at 288°C is greater at low strain rates (Chopra & Muscara, 2002).

- ✓ LWR environments

The fatigue life of austenitic strainless steels decreases in LWR environments with primarily strain rate and temperature. Dissolved oxygen (DO) content in the water and material heat treatment may also influence fatigue life.

2.3.3 Critical parameter that influence the fatigue life

The critical parameters that influence fatigue life and the threshold values of these parameters for environmental effects are:

- ✓ **Strain amplitude** - a slow strain rate applied during the tensile-loading cycle is primarily responsible for environmentally assisted reduction in fatigue life.
- ✓ **Hold-Time Effects** - Environmental effects on fatigue life occur primarily during the tensile-loading cycle and at strain levels greater that the threshold value. Consequently, loading and environmental conditions during the tensile-loading cycle, e.g., strain rate, temperature, and DO level are important for environment assisted reduction of the fatigue lives of these steels.
- ✓ **Strain rate** - in LWR environment fatigue life decreases logarithmically with decreasing strain rate.
- ✓ **Dissolved Oxygen** - the fatigue lives of austenitic strainless steels decrease significantly in low-DO water; the effect is greater at low strain and high temperature, on the other hand, high water environmental effects on the fatigue lives are not well known.
- ✓ **Temperature** - the change in fatigue lives of austenitic strainless steels suggest a threshold temperature of 150°C, above which the environment decreases fatigue life in low-DO water if the strain rates is below the threshold of 0.4%/s. In the range of 150-325°C, the logarithm of fatigue life decreases linearly with temperature. Only moderate decrease in life is observed in water at temperature below the threshold value of 150°C.
- ✓ **Flow rate**, it is generally recognized that flow rate most likely has a significant effect on the fatigue life of materials because it causes differences in local environmental conditions, however the effect of flow rate on the fatigue life of austenitic strainless steels has not been evaluated. Cast Stainless Steels indicates that in air, the fatigue lives of cast CF-8 and CF8M stainless steels are like that of wrought austenitic stainless steels (Chopra & Muscara, 2002).

2.3.4 Mechanism of fatigue crack initiation

The fatigue life of a material is defined as the number of cycles necessary to form an engineering crack. Fatigue crack has been divided into two stages:

- ✓ An initiation stage that involves the growth of microstructurally small cracks.
- ✓ Propagation stage that involves the growth of mechanically small cracks.

Researches indicated that decreased in the fatigue life of 316L steels are caused primarily by the effects of environment on the growth of microstructurally small cracks and, to a less extent on enhanced growth rates of mechanically small cracks. In low-DO water, fatigue cracks are always straight and normal to the stress axis, whereas in airs or high-DO water they follow certain crystallographic features. However, the morphology of crack growth into the material is similar in both air and water (Chopra & Muscara, 2002).

2.4 Reliability

Reliability, as a human attribute, has been praised for a very long time. For technical systems, however, the reliability concept has not been applied for more than some 60 years. It emerged with a technological meaning just after World War I and was then used in connection with comparing operational safety of one-, two-, and four-engine airplanes. Reliability was measured as the number of accidents per hour of flight time. At the beginning of the 1930s, Walter Shewhart, Harold F. Dodge, and Harry G. Romig laid down the theoretical basis for utilizing statistical methods in quality control of industrial products. Such methods were, however, not brought into use to any great extent until the beginning of World War II. Products that were composed of many parts often did not function, even though they were made up of individual high-quality components.

During World War II a group in Germany was working under Wernher von Braun developing the V- 1 missile. After the war, it was reported that the first 10 V- 1 missiles were all fiascos. Despite attempts to provide high-quality parts and careful attention to details, all the first missiles either exploded on the launching pad or landed “too soon”. Robert Lusser, a mathematician, was called in as a consultant. His task was to analyze the missile system, and he quickly derived the product probability law of series components. This theorem concerns systems functioning only if all the components are functioning and is valid under special assumptions. It says that the reliability of such a system is equal to the product of the reliabilities of the individual components which make up the system (Høyland & Rausand, 1994).

2.4.1 Reliability concept

The reliability was defined until the 1960s as “the probability that an item will perform a required function under stated conditions for a stated period. Some authors still prefer this definition, for example, Smith (1997) and Lakner and Anderson (1985). But, there is a general definition of reliability given in standards like IS0 8402 and British Standard BS 4778: Reliability is the ability of an item to perform a required function, under given environmental and operational conditions and for a stated period (ISO 8402).

- ✓ The term “item” is used here to denote any component, subsystem, or system that can be considered as an entity. A required function may be a single function or a combination of functions that is necessary to provide a specified service;
- ✓ All technical items (components, subsystems, systems) are designed to perform one or more (required) functions. Some of these functions are active and some functions are passive. Containment of fluid in a pipeline is an example of a passive function. Complex systems (e.g., an automobile) usually have a wide range of required functions. To assess the reliability (e.g., of an automobile), we must first specify the required function(s) we are considering.

For a hardware item to be reliable, it must do more than meet an initial factory performance or quality specification, it must operate satisfactorily for a specified period of time in the actual application for which it is intended (Høyland & Rausand, 1994).

2.4.2 Mathematical definition for reliability

The state of an item at time may be described by the state variable.

$$x(t) = \begin{cases} 1 & if\ the\ item\ is\ functioning\ at\ time\ t \\ 0 & if\ the\ item\ is\ in\ a\ failed\ state\ at\ time\ t \end{cases} \qquad (2.1)$$

By the *time to failure* of an item mean the time elapsing from when the item is put into operation until it fails for the first time. It is therefore natural to interpret the time to failure as a random variable. If the time to failure is continuously distributed with probability density function $f(t)$ and distribution function $F(t)$, the eq. (2.2) denotes the probability that the item fails within the time interval.

$$F(t) = Pr(T \leq t) = \int_0^t f(u)du \quad for\ t > 0 \tag{2.2}$$

The probability density function $f(t)$ is defined as:

$$f(t) = \frac{d}{dt}F(t) = \lim_{\Delta t \to \infty} \frac{F(t+\Delta t) - F(t)}{\Delta t} \tag{2.3}$$

- ✓ Reliability function

The reliability function of an item is defined by:

$$R(t) = 1 - F(t) = Pr(T > t)\ for\ t > 0 \tag{2.4}$$

- ✓ Failure rate function

The probability that an item will fail in the time interval when we know that the item is functioning at time is:

$$Pr(t < T \leq t + \Delta t |\ T > t) = \frac{\Pr(t < T \leq t + \Delta t)}{Pr(T > t)} = \frac{F(t + \Delta t) - F(t)}{R(t)} \tag{2.5}$$

2.5 Reliability test method

Reliability testing has become a useful way to meet and exceed customer demand for high quality and reliable products. It usually involves simulation of conditions under which the item will be used during its lifespan. Reliability does not compare the product to some predefined specifications, such as the case with quality assurance, but rather investigates the performance over a predefined period of time (Antonitsin, 2009).

Different types of reliability test and screens are used in different phases of the product development/production processes. Table 1 outlines the kinds of reliability test done at different stage of product design and production.

Table 1: Reliability test (Meeker & Escobar, 1998)

Product Design		**Product production**
Qualification testing of material and components	Prototype testing of System and Subsystem	Production and post-production screens for systems and subsystems
Accelerated degradation test	Robust Design Test STRIFE test	Component Certification burn-in
Accelerated life tests	Test-and-Fix for reliability growth	Environmental stress screening

Generally, there is a need to do these tests as quickly as possible, and methods have been developed to accelerate all of these different types of tests and scream. The focus in this dissertation is the accelerated life tests that are done during product design to assist reliability and quantify the use of proposed materials and components (Meeker & Escobar, 1998).

2.6 Accelerated life test

The term “acceleration” has many different in reliability, but it generally implies making “time” (on whatever scale is used to measure device or component life) go more rapidly, so that reliability information can be obtained more quickly. The Accelerated testing (AT) is a way to predict the life at use conditions. For the test it is necessary the extrapolation of the time and the accelerating and it needs to be justified on the basis of physically motivated models, for it the statistic have developed important contributions with stochastic models for AT data.

2.6.1 Quantitative versus Qualitative Accelerated test

Within the reliability discipline, the term “accelerated test” is used to describe two different kinds of useful tests that have different purposes. To distinguish between these, the terms “quantitative accelerated tests” (QuanAT) and “qualitative accelerated tests” (QualAT) are sometimes used. A QualAT has purpose to identify product weaknesses caused by flaws in the product’s design or manufacturing process. The QuanAT tests units at combinations of higher than-usual levels of certain accelerating factors. The purpose of a QuanAT is to obtain information about the failure-time distribution or degradation distribution at specified “use” levels of these factors. Generally, failure modes of interest are known ahead of time, and there is some knowledge available that describes the relationship between the failure mechanism and the accelerating factors (either based on physical/chemical theory or large amounts of previous experience with similar tests) that can be used to identify a model that can be used to justify the extrapolation (Escobar & Meeker, 2007).

2.6.2 Method of Acceleration

There are different methods of accelerating a reliability test (Escobar & Meeker, 2007):

- ✓ Increase the use rate of the product - This method is appropriate for products that are ordinarily not in continuous use. For example, the median life of a bearing for a certain washing machine agitator is 12 years, based on an assumed use rate of 8 loads per week. If the machine is tested at 112 loads per week (16 per day), the median life is reduced to roughly 10 months, under the assumption that the increased use rate does not change the cycles to failure distribution. Also, because it is not necessary to have all units fail in a life test, useful reliability information could be obtained in a matter of weeks instead of months.
- ✓ Increase the intensity of the exposure to radiation - Various types of radiation can lead to material degradation and product failure. For example, organic materials (ranging from human skin to materials like epoxies and polyvinyl chloride or PVC) will degrade when exposed to ultraviolet (UV) radiation. Electrical insulation exposed to gamma rays in nuclear power plants will degrade more rapidly than similar insulation in similar environments without the radiation. Modeling and acceleration of degradation processes by increasing radiation intensity is commonly done in a manner that is similar to acceleration by increasing use rate.
- ✓ Increase the aging rate of the product - Increasing the level of experimental factors like temperature or humidity can accelerate the chemical processes of certain failure mechanisms such as chemical degradation (resulting in eventual weakening and failure) of an adhesive mechanical bond or the growth of a conducting filament across an insulator (eventually causing a short circuit).
- ✓ Increase the level of stress (e.g., amplitude in temperature cycling, voltage, or pressure) under which test units operate - A unit will fail when its strength drops below applied stress. Thus, a unit at a high stress will generally fail more rapidly than it would have failed at low stress.

- ✓ Combinations of these methods of acceleration are also employed - Factors like voltage and temperature cycling can both increase the rate of an electrochemical reaction (thus accelerating the aging rate) and increase stress relative to strength. In such situations, when the effect of an accelerating variable is complicated, there may not be enough physical knowledge to provide an adequate physical model for acceleration (and extrapolation). Empirical models may or may not be useful for extrapolation to use conditions (Escobar & Meeker, 2007).

It is useful to distinguish among ATs on the basis of the nature of the response.

- ✓ Accelerated Binary Tests (ABTs). The response in an ABT is binary. That is, whether the product has failed or not, is the only reliability information obtained from each unit.
- ✓ Accelerated Life Tests (ALTs). The response in an ALT is directly related to the lifetime of the product. Typically, ALT data are right-censored because the test is stopped before all units fail. In other cases, the ALT response is interval-censored because failures are discovered at particular inspection times.
- ✓ Accelerated Repeated Measures Degradation Tests (ARMDTs). In an ARMDT, one measures degradation on a sample of units at different points in time. In general, each unit provides several degradation measurements. The degradation response could be actual chemical or physical degradation or performance degradation (e.g., drop in power output).
- ✓ Accelerated Destructive Degradation Tests (ADDTs). An ADDT is similar to an ARMDT, except that the measurements are destructive, so one can obtain only one observation per test unit.

These different kinds of ATs can be closely related because they can involve the same underlying physical/chemical mechanisms for failure and models for acceleration. They are different, however, in that different kinds of statistical models and analyses are performed because of the differences in the kind of response that can be observed. Many of the underlying physical model assumptions, concepts and practices are the same for ABTs, ALTs, ARMDTs and ADDTs. There are close relationships among models for ABT, ALT, ARMD and ADD data. Because of the different types of responses, however, the actual models fitted to the data and methods of analysis differ. In some cases, analysts use degradation-level data to define failure times. For example, turning ARMDT data into ALT data generally simplifies analysis but may sacrifice useful information. An important characteristic of all ATs is the need to extrapolate outside the range of available data: tests are done at accelerated conditions, but estimates are needed at use conditions. Such extrapolation requires strong model assumptions (Escobar & Meeker, 2006).

2.6.3 Acceleration models

Interpretation of accelerated test data requires models that relate accelerating with factors like, temperature, voltage, pressure, and size to time acceleration. For testing over some range of accelerating factors, one can fit model to the data to describe the effect that the factors have on the failure causing processes. The general idea is at high levels of the accelerating variable(s) to speed up failure processes and then to extrapolate to lower levels of the accelerating factors(s). For some situation, a physically reasonable statistical model may allow such extrapolation.

- ✓ Physical acceleration models

For well-understood failure mechanisms, one may have a model based on physical/chemical theory that describes the failure-causing process over the range of the data and provides extrapolation to use conditions. The relationship between accelerating factors and the actual failure mechanism is usually extremely complicated. Often, however, one has a simple model that adequately describes the process.

- ✓ Empirical acceleration models

When there is little understanding of the chemical of physical processes leading to failure, it may be impossible to develop a model based on physical/chemical theory. An empirical model may be the only alternative. An empirical model may provide an excellent fit to the available data but may provide nonsense extrapolations. In some situations, there may be extensive empirical experience with particular combinations of factors and failure mechanisms and this experience may provide the needed justification for extrapolation to use conditions (Meeker & Escobar, 2006).

2.7 Experimental design

Ronald A. Fisher invented the experiments design in the 1920s and 1930s at Rothamsted Experimental Station, agricultural research station 25 miles north of London. Fisher proposed the Design of Experiments (DOE) based on experiments with natural fluctuations such as soil conditions, temperature, and rainfall. Statistical design of experiments refers to the process of planning the experiment so that appropriate data will be collected and analyzed by statistical methods, resulting in valid and objective conclusions. The statistical approach to experimental design is necessary if we wish to draw meaningful conclusions from the data. When the problem involves data that are subject to experimental errors, statistical methods are the only objective approach to analysis.

The three basic principles of experimental design are randomization, replication, and blocking.

Randomization is the cornerstone underlying the use of statistical methods in experimental design, in an experimental design, it means that both the allocation of the experimental material and the order in which the individual runs of the experiment are to be performed are randomly determined. Statistical methods require that the observations (or errors) be independently distributed random factors. Randomization usually makes this assumption valid. By properly randomizing the experiment, we also assist in "averaging out" the effects of extraneous factors that may be present.

Replication is an independent repeat run of each factor combination. Replication has two important properties. First, it allows the experimenter to obtain an estimate of the experimental error. This estimate of error becomes a basic unit of measurement for determining whether observed differences in the data are statistically different. Second, if the sample mean is used to estimate the true mean response for one of the factor levels in the experiment, replication permits the experimenter to obtain a more precise estimate of this parameter.

Blocking is a design technique used to improve the precision with which comparisons among the factors of interest are made. Often blocking is used to reduce or eliminate the variability transmitted from nuisance factors that is, factors that may influence the experimental response but in which we are not directly interested (Montgomery, 2012).

2.7.1 Guidlines for designing experiments

To use the statistical approach in designing and analyzing experiments, it is necessary for the experiment to have a clear idea in advance of what is to be studied, how the data are to be collected, and at least a quantitative understanding of how these data are to be analyzed, the guidelines are: Recognition of a statement of the problem, Choice of factors, levels and ranges, Selection of the response variable, Choice of an experimental design, Performing the experiment, Statistical analysis and Conclusion.

1. Recognition of a statement of the problem - It is necessary to develop all ideas about the objectives of the experiment. Usually it is important to solicit input from all concerned parties: engineering, quality assurance, manufacturing, marketing, management, the costumer, and operating personal. For that reason, a team approached to designing an experiment is recommended.

   It is usually helpful to prepare a list of specific problems that are to be addressed by the experiment. A clear statement of the problem often contributes substantially to better understanding of the phenomenon being studied and the final solution of the problem.

2. Choice of factors, levels and ranges - When considering the factors that may influence the performance of a process or system, the experimenter usually discovers that these factors can be classified as either potential design factors or noise factors. The potential design factors are those factors that the experiment may wish to vary in the experiment. Often, we find that there are a lot potential design factors and often some classification of them is helpful. Some helpful classification is, design factors, held constant factors, and allowed to vary factors. The design factors are the factors actually for experiment. Held constant factors are factors that may exert some effects on the response, but for the purpose of the present experiment are not of interest, so they will be held at a specific level. Nuisance factor are the factors that could be influencing the variable answer but not necessary taking into account.

3. Selection of the response variable - In selecting the response variable, the experimenter should be certain that this variable really provides useful information about the process under study. Most often, the average or standard deviation (or both) of the measured characteristic will be the response variable. Multiple responses are not unusual. Gauge capability is also an important factor because if it is inadequate only

relatively large factors effect will be detected by the experiment or perhaps additional replication will be required.

4. Choice of an experimental design. If the pre-experimental planning activities above are done correctly, this step is relatively easy. The choice of the experimental design involves the consideration of sample size (number of replicates), the selection of the suitable run order for the experimental trials, and the determination of whether or not blocking or another randomization restriction are involved.
5. Performing the experiment - When running the experiment, it is vital to monitor the process carefully to ensure that everything is being done according to the plane. Errors in the experimental procedure at this stage will usually destroy experimental validity. It is easy to underestimate the logistical and planning aspect of running a designed experiment in a complex manufacturing or research and development environment.
6. Statistical analysis of the data - Statistical methods should be used to analyze the data so that results and conclusion are objective rather than judgmental in nature. If the experiment has been designed correctly and if it has been performed according to the design, the statistical methods required are not elaborate.
7. Conclusion and recommendations - Once the data have been analyzed, the experimenter must draw practical conclusions about the results and recommend a course of action. Graphical methods are often useful in this age particularly in presenting the results to others (Montgomery, 2012).

2.7.2 Taguchi method

Ealey Lance (1994) says that the main trust of Taguchi's techniques is the use of parameter design, which is an engineering method for product or process design that focuses on determining the parameter (factor) settings producing the best levels of a quality characteristic (performance measure) with minimum variation. Taguchi designs provide a powerful and efficient method for designing processes that operate consistently and optimally over a variety of conditions. To determine the best design, it requires the use of a strategically designed experiment, which exposes the process to various levels of design parameters.

Experimental design methods were developed in the early years of 20th century and have been extensively studied by statisticians since then, but they were not easy to use by practitioners. Taguchi's approach to design of experiments is easy to be adopted and applied for users with limited knowledge of statistics; hence it has gained a wide popularity in the engineering and scientific community. Taguchi specified three situations: Larger the better (for example, agricultural yield); Smaller the better (for example, carbon dioxide emissions); and On-target, minimum-variation (for example, a mating part in an assembly). See Figure 5, that represent how the product, process, and systems are directly influenced by the control, noise and signal samples and those implicates later a response variable.

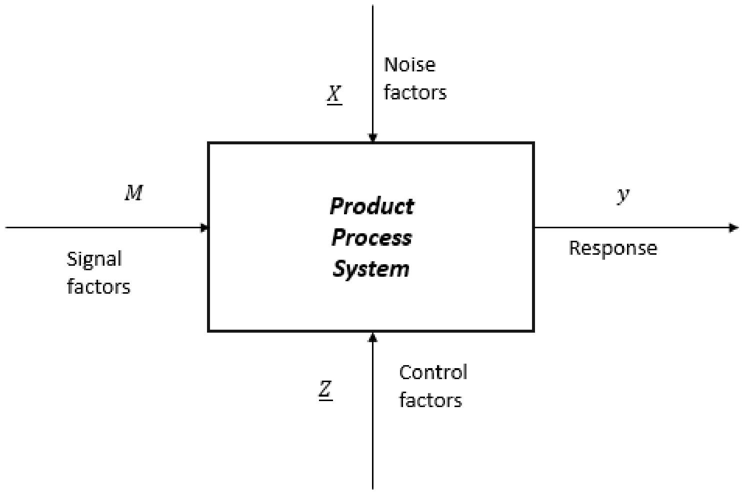

Figure 5: Parameter diagram of a Product/Process/System

Taguchi realized that the best opportunity to eliminate variation is during the design of a product and its manufacturing process. Consequently, he developed a strategy for quality engineering that can be used in both contexts. The process or product has three stages of development: System design, Parameter design, and tolerance design The first one involve creativity and innovation, the second refers to robustification and third the reduction and control of variation in the critical dimension (Karna & Sahai, 2012).

The effect of many different parameters on the performance characteristic in a process can be examined by using orthogonal arrays of experimental design proposed by Taguchi (see Table 2) the possible dimension of the samples according with the levels and the numbers of factors. Once the parameters affecting a process that can be controlled have been identified, the levels at which these parameters should vary must be determined. Determining what levels of a variable to test requires an in-depth understanding of the process, including the minimum, maximum, and current value of the parameter. If the difference between the minimum and maximum value of a parameter is large, the values being tested can be further apart or more values can be tested. If the range of a parameter is small, then less value can be tested or the values tested can be closer together.

Table 2: Array selector

		Number of parameter(p)								
		2	3	4	5	6	7	8	9	10
No. of level	2	L4	L4	L8	L8	L8	L8	L12	L12	L12
	3	L9	L9	L9	L18	L18	L18	L18	L27	L27
	4	L'16	L'16	L'16	L'16	L'32	L'32	L'32	L'32	L'32
	5	L25	L25	L25	L25	L25	L50	L50	L50	L50

The Taguchi method is a powerful tool for designing high quality systems. To increase the experimental efficiency, the L18 mixed orthogonal table in the Taguchi quality design has been used to determine the significant factors. In experiments, influential machining parameters are selected, such as cutting tools of different materials, depth of cut, cutting speed, feed rate, working temperature and ultrasonic power.

In the parameter design stage Taguchi makes use of experiment design, using the orthogonal arrays, and signal to noise ratios to determine the optimal parameter settings. The Signal-to-Noise ratios (S/N) (see Eq. 2.6), which are log functions are based on “Orthogonal array”. The use of experiment design and signal /noise ratios is to determine the optimal parameter which gives reduced “variance “for the experiment, defining the “optimum settings “of control parameters. Equation 2.6 is used when the characteristics of the response variable is of the type large the better. There are other equations for response variable of the type the smaller the better or nominal is better (Karna & Sahai, 2012).

$$(S/N)_i = -10log\left[\frac{1}{n}\sum_{j=1}^{n}\frac{1}{Y_{ij}^2}\right] \quad (2.6)$$

i is the number of a trial;

Y is the measured value of quality characteristic for the i^{th} trial

j^{th} is the number of experiment;

n is the number of repetition for the experimental combination

2.8 Accelerating testing using experimental design

This section explores experimental design concept, including concepts such as planning and design, but directly focused on the application of accelerate tests.

2.8.1 Test planning

The test planning plays a relevant role for an experiment, a relevant step in the proper development of it is the determination of the noise (see item 2.6). The stress stress is the means of acceleration for the material in order to have faster results, thus the following must be identified:

- ✓ The stresses the unit are exposed to, under use conditions;
- ✓ Which of those stresses most probably degrade the unit's performance;
- ✓ Which of the identified stresses can be effectively controlled under a test environment to be measured.

This is important because failures can be propagated to very high levels of stress but also in very low levels of stress (MoKinney, 1993).

There are three important aspects to the combined test and the stress levels:

- ✓ The overall range of the test stresses and the number of test levels;
- ✓ The consideration of the eventual customer;
- ✓ The process being formulated as a traditional designed experiment.

2.8.2 Design phase

The design phase of accelerated testing needs to remains free of typical accelerated testing shortcomings, such as assuming a specific time-to-failure distribution, assuming a stress/performance relationship function (e.g., the Arrhenius relationship), and utilizing lengthy extrapolations. A Design of Experiments approach, using a combined stress environment will avoid these pitfalls (Mashhadi, 1992).

Advantages

- ✓ The use of accelerated life testing reduces the test time significantly while the use of factorial design, by testing the effect of several factors simultaneously, provides the maximum possible information from a minimum number of tests. Combining these two methods is to benefit from the advantages of both methods;
- ✓ The combination between these two methods increase the efficiency in an exploratory improvement investigation, when the factorial design is the main intention and the other is for verification of reliability, when accelerated testing is called for.
- ✓ When verification via accelerated testing is the main purpose, a combination with factorial design serves to study the effect of disturbing factors and other interesting factors. Information can be obtained concerning the effect of these factors, interactions between them, and the interaction between each factor and the stress factor (Mashhadi, 1992).

Disadvantage

- ✓ In some cases, the failure mechanism under study changes as the stress is increased. This is one of the main reasons for some people to question the use of accelerated life testing. However, application of factorial designs at increased stress levels can be used to identify such a change (Mashhadi, 1992).

2.9 Research's Background

2.9.1 Environmental Effects on Fatigue Crack Initiation in Piping and Pressure Vessel Steels (O. Chopra, 2001)

The Argonne National Laboratory performed a study on fatigue of carbon and low-alloy steel and austenitic SSs in LWR environment. The objective was to evaluate the effects of the fatigue S-N life in the factors: steel type, strain range, strain rate, temperature, content in carbon and low–alloy steels orientation, and DO (Dissolved Oxygen) level in water . The time was measured in the number of cycles (N) to failure and S (Stress). In order to calculate the fatigue, statistical model is used in function of the type of Material, Loading and environment. This research also presented differenced investigations in the nuclear fatigue failures and later on discusses about the effect in crack initiation.

For the American Society of Mechanical Engineers (ASME), two methods are used based on statistical models: the design fatigue method and the fatigue life correlation factor methods. Even being different between each other, both methods are conclusive and can be used to environmental effects analyses.

The results for the ASME Code suggest that fatigue life is associated with the factor material and loading conditions are reasonable.

3. SCK•CEN EXPERIMENTS IN FATIGUE CRACK INITIATION

This chapter presents a description of SCK•CEN and an overview of MYRRHA reactor and some research results developed inside the company.

3.1 SCK•CEN: the Belgian Nuclear Research center

The Belgian Nuclear Research Centre was founded in 1952. This gave the Belgian academic and industrial world access to the worldwide development of nuclear energy. Ever since SCK•CEN has been playing a pioneering role with unique achievements and groundbreaking work in nuclear science and technology. Today SCK•CEN, with laboratories in Mol, Belgium and a registered office in Brussels, is one of the largest research centers in Belgium.

SCK•CEN conducts fundamental and applied research at an advanced scientific level and in an international context. The objective is to work on issues that are important to society today and tomorrow (Bausart et al., 2011).

SCK•CEN has three scientific institutes:

- ✓ The Institute for Nuclear Materials Science, carries out research on materials and fuels used in present and future nuclear installations;
- ✓ The Institute for Advanced Nuclear Systems, It supports the nuclear industry and authorities on a national and international level;
- ✓ The Institute for Environment, Health and Safety studies the behavior of radioactive substances in the biosphere (air, water, soil, plants, etc.) and geosphere (subterranean clay layers, groundwater, etc.) and evaluates the impact of radiation on mankind and the environment (Bausart et al., 2011).

3.1.1 MYRRHA

MYRRHA is an Accelerator Driven System (ADS) for transmuting long-lived radioactive waste, which will contribute to addressing key issues for our future world. In a rapid growing world, the needs increase in a huge speed, in consequence it is necessary to provide advanced technology, and MYRRHA is designed with the objective to answer the technology demand through its flexible fast spectrum research reactor. This reactor has multiple applications of which the following stand out: Sustainable energy, Enabling technologies for renewable energies, Health care, Science.

An AD (Accelerator Driven) - sometimes also called an energy amplifier or hybrid system - is an alternative concept to the critical nuclear reactor. The idea was first proposed by Nobel Prize laureate E.O. Lawrence in the 1950's and revived by another Nobel Prize laureate C. Rubbia in 1993 after recent major advances in accelerator technology. The main advantage of ADS (Accelerator Driven System) is its non-critical fission core, i.e. a core that cannot on its own sustain the fission chain reaction (Van Tichelen et al., n.d.).

The design of the reactor MYRRHA ADS currently performing is based on a pool type configuration with a Standing Vessel (Figure 6). It allows also having an internal interim storage easing the fuel handling (Abderrahim & Malambu, 2005).

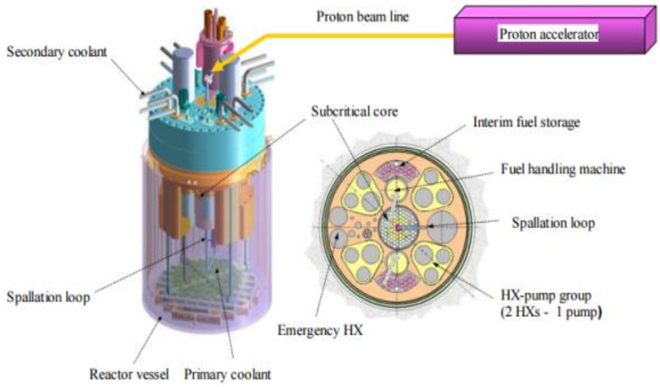

Figure 6: Parts of MYRRHA

The natural circulation for the residual heat removal in case of loss of flow (LOF) and loss-of-heat-sink (LOHS) is demonstrated to be feasible, particularly with the large thermal inertia that is also an argument in favor of this design. With an emergency cooling system based on natural circulation both on primary and secondary sides, a long-term core cool ability can be ensured even in situation of complete power loss. The core pool contains a fast-spectrum sub-critical core loaded with typical fast reactor hexagonal assemblies cooled by LBE. The three central hexagons are left free for housing the spallation module. The spallation target circuit is fully immersed in the reactor pool and thermally interlinked with the core, but its liquid metal is physically separated from the core coolant (Abderrahim, Sobolev, & Malambu, 2005).

3.2 Research's projects in SCK•CEN

In order to be up with the development SCK•CEN is always working in new research, this section has two projects related with the topic in this dissertation made by SCK•CEN group and finally is performed a critical analysis of those researchers.

3.2.1 The Fatigue Behavior of T91 and 316L Steels

In October 2012, Pierre Marmy and Xing Gong wrote a paper "*The Fatigue Behavior of T91 and 316L Steels"*, to describe the fatigue on the steels T91 and 316L two of the commons materials used in SCK•CEN to study LBE coolant (Marmy & Gong, 2012).

For the fulfillment of the objectives a strategic data collection was made and clearly explained. To select the factors, next conditions were considered: specimen, geometry and test machine, the test conditions (environment, mechanical conditions), and the material state (chemical analysis, heat treatments). The factors on study were: temperature, strain rate, hold times, chemical analysis, environmental medium.

In the paper conclusions, it is possible to see that, crack initiation of T91 and 316L steels occurs at the surface, at persistent slip band (PSB), and in rare case crack initiates at inclusion and grain boundary. About the parameters, the conclusion was that:

- ✓ **Temperature**. The fatigue endurance for both steels, decreases with the increase of the temperature.
- ✓ **Hold times**. At elevate temperature, hold time has a detrimental effect on the fatigue, on the other hand 316L is more susceptible to tension hold.
- ✓ **Strain rate.** It has a weak effect on fatigue endurance for T91 steel for all levels of the temperature, in case of 316L the occurrences of DSA (Dynamic strain ageing) became sensitive to strain rate strong when the temperature is between 400-600ºC.
- ✓ **Environment**. LBE reduces the continuous fatigue life in T91 steel. For 316L steel, it affects negatively the fatigue endurance at 260ºC.

3.2.2 Influence of LBE coolant on the fatigue properties of 316L steel

A study was performed in 2015 by Pierre Marmy and Xing Gong, that addressed the "*Influence of LBE on the fatigue properties of 316L steel*", which analyzed the LBE coolant and the characteristics of 316L that have been found to have great compatibility in that environment.

(Marmy & Gong, 2015) used a machine to perform the tests at SCK•CEN capable to produce fatigue results in steels, in the whole operating parameter range of the MYRRHA reactor. This novel fatigue test system, called LIMETS 3 has been described and validated. A innovation of this facility is an original mechanical extensometer that is capable of directly measuring the strain at the gauge length of specimens submersed in LBE at temperatures up to 500°C.

The conclusion obtained with the analysis shows that the LBE pre-exposure time has no clear effect on fatigue life, and that compared to vacuum, a reduction of the fatigue life in LBE at 300°C was observed. However, the fatigue life in LBE is comparable with that in air.

3.2.3 Critical analysis of the performed works

The works performed by Pierre Marmy and Xing Gong described use similar methodologies for the realization and analysis of projects to study LBE in SCK•CEN. The conclusions in the articles are important and show results that can be used for decisions in the future. The authors describe clearly the data collection related with the step taken before and during the procedure, and present specific graphic for later describing the information collected.

However, the papers do not show relevant procedure, statistically in terms of design and planning regarding samples sizes and repetitions. Everything that deals with the collection, processing, interpretation and presentation of data belongs to the domain of statistics, and so does the detailed planning of that precedes. To develop a good investigation through statistic it is necessary to find answers to the following questions (Isotalo, 2001): What kind and how much data need to be collected? How should we organize and summarize the data? How can we analyze the data and draw conclusions from it? How can we assess the strength of the conclusions and evaluate their uncertainty.

To answer the questions, statistic as a methodology for interpreting and drawing conclusions from collected data provides different techniques as experimental design (simple experiments, by blocks, factorial, among others) (see section 2.6).

Statistic allows the use of different techniques in order to infer about the population on study, the two-researches reviewed, show the use of graphics to do a visualization of the data behavior. However, more methodologies can be used according to statistic, as for example: calculation of indicators and the use of the inferential statistic use of models that can bring more information about the sample and if it allows, subsequently, on the study population.

4. STATISTICAL ANALYSIS OF DATA FROM SCK•CEN

This chapter presents the statistical analysis for two set of data in which deceptive and inferential statistic are used as an alternative to the methodologies used previously in SCK•CEN.

4.1 Practical experimental method of realization

According with Marmy Pierre (2017), the machine used for the study in SCK•CEN (See Figure 7) is a home developed system based on a MTS hydraulic cylinder with adequate software which can apply the force to the specimen. The test is strain controlled, through a strainmeter placed on the gauge length of the specimen, which evaluates the required strain applied by the movement of the cylinder. The command signal is a triangle and, its amplitude and frequency can be controlled. The test is finished when the force signal decreases more than a pre-set level.

The test is run into a vessel filled with the LBE, which is purified by a gas flow of Ar - 5% H2 mixture. This way, the O (Oxygen) is consumed by the hydrogen and exit the metal into the gas plenum. This process is very slow and requires 1 or 2 days. Currently, it can be chosen between oxygen saturated (the cleaning of is turned) or oxygen free where the liquid metal becomes corrosive (no oxide layers can be formed on the specimen surface).

Figure 7: Testing machine by Marmy Pierre (2017)

4.2 Statistical data analysis for the fatigue in LBE environment at 300 and 400°C

4.2.1 Test conditions

The studied 316L material is from a hot rolled plate of 6000x2000x15mm produced by the group Arcelor INDUSTEEL, in Charleroi, with the number of identification 261612/1 from heat number 55801.

The material has received a standard heat treatment: it was first solution-annealed at 1050°C for 15 min and subsequently water quenched. The chemical composition of the steel is given in Table 3 in which are represented the proportion of each element in the composition of the steel.

Table 3: Chemical composition of 316L

C	Cr	Ni	Mo	Mn	V	Si	Nb	N	Fe
0.016	16.82	10.17	2.086	1.84	-	0.641	-	0.025	Bal.

The low cycle fatigue tests were carried out on cylindrical specimens with a gauge length of 7 mm and a diameter of 3.2 mm. The surface of the fatigue specimens was first polished using alumina oxide paste to eliminate the machining markings at the surface and then diamond paste down to 3 μm for fine polishing. Fatigue tests were conducted with a servo-hydraulic MTS machine under strain control. The total strain ranges imposed to the specimens varied from 0.66% to 2.44%. Most of the tests were carried out at a constant strain rate of around 4.5×10^{-3} s^{-1}. Two specimens were tested at different strain rates, i.e. 1.43×10^{-2} s^{-1} and 10^{-3} s^{-1} to check the impact of strain rate on material behavior and fatigue endurance. The fatigue tests were performed in LBE at 300°C and 400°C. During each test, the oxygen concentration in LBE was monitored by the oxygen sensors.

Two series of reference tests have been conducted: one in air at room temperature and the second under vacuum (2×10^{-4} and 2×10^{-5} mbar) at 300°C (Marmy & Gong, 2015).

The results obtained are presented in Table 4, which is data taken from Marmy & Gong (2015):

- ✓ **Specimen** - represents the element object of the study;
- ✓ **Steel**- represents the name of the steel for the experiment that, in this study, is always the same;
- ✓ **Test Temperature**- represents the temperature used in the experiment in Celsius °C;

- ✓ **Environment** - indicates the type of coolant that in this case is LBE;
- ✓ **C/wt** - is the oxygen wt;
- ✓ **Pre-exposure** - represents exposition of the specimen to LBE for the time shown in the table, at the temperature indicated. This is required to lower the oxygen concentration until the level is low enough, and the time is given in hours and the temperature in grades Celsius;
- ✓ **Strain rate** - indicates the speed that the deformation occurs measured in seconds;
- ✓ $\Delta\boldsymbol{\varepsilon_t}$- indicates the fatigue in term of the total strain amplitude (No Units);
- ✓ $\Delta\boldsymbol{\varepsilon_e}$- indicates the fatigue in term of elastic strain amplitude (No Units);
- ✓ $\Delta\boldsymbol{\varepsilon_p}$- is the representation of the fatigue in term of the plastic strain amplitude (No Units);
- ✓ **Stress range** - is the difference between the maximum and minimum stress in one fatigue test cycle in megapascal (MPa);
- ✓ $\boldsymbol{N_f}$- The number of cycles until the failure;
- ✓ $\boldsymbol{N_a}$ - The cycles required to form an already macroscopic crack;
- ✓ $\boldsymbol{N_f - N_a}$- The function of strain amplitude.

For the statistical analysis, the fourteen columns in Table 4 are not all necessary, because between them there are factors: constants, or represented through another factor (dependent factors).

The factors "Steel and Environment" remain constant throughout the tests. The factors $\Delta\varepsilon_t$ is calculated through $\Delta\varepsilon_e$ and $\Delta\varepsilon_p$, so to reduce the number of factors in the analysis $\Delta\varepsilon_t$ is going to be used. In the case of the Stress range and the Strain rate, the last one is considered because one is represented through the other. Finally, among the factors N_f, N_a, $N_f - N_a$ in which, the last one is a dependent factor, N_a have a secondary relevance on the objective, N_f is in this case the factor to be used because of its relevance for this project.

Table 4: Data from Marmy & Gong (2015)

Specimen ID	Test Temperature	C/wt	Pre-exposure	Strain rate	$\Delta\varepsilon_t$	$\Delta\varepsilon_p$	$\Delta\varepsilon_e$	Stress range	N_f	N_a	$N_f - N_a$
2F28	300	0,00000198	1h,230 to 300	0,00491	1,09	0,81	0,28	476,3	7390	7300	90
2F30	300	0,00000466	1h,230 to 300	0,00441	1,96	1,5	0,46	694,2	1078	1064	14
2F31	300	0,00000541	1h,230 to 300	0,00476	0,74	0,53	0,21	451	16456	16293	163
2F33	300	0,00000583	1h,230 to 300	0,00402	0,59	0,31	0,28	452,1	108556	107581	975
2F37	300	0,00000521	1h,230 to 300	0,00422	0,9	0,64	0,26	440,2	11463	11200	263
2F60	300	0,000004	72h,400	0,0045	1,08				4200		
2F61	300	0,000008	26h,400	0,0045	0,78				10775		
2F39	400	0,000000014	70h,400	0,001	1,19	0,78	0,41	551,8	6004	5807	197
2F40	400	0,000000024	70h, 400 to 410	0,00143	1,19	0,86	0,33	536	4788	4720	68
2F41	400	0,000000021	542h,400	0,0045	1,2	0,86	0,34	541,61	8465	8380	85

4.2.2 Statistical analysis

To preliminary explore data; Table 5 presents the Mean and standard deviation of the Oxygen, Strain rate, total strain amplitude, number of cycle until the failure and the number of cycles until the formation of macroscopic crack. Those factors are interesting to analyze because according to the projects developed before, they have direct influences on fatigue crack initiation.

Table 5: Descriptive analysis

Temperature	Trend measures	C/wt	Strain rate	$\Delta\varepsilon_t$	N_f	N_a
300	Mean	5,0129E-06	0,00447429	1,02	22845,4	28687,6
300	Deviation Standard	1,8319E-06	0,00030221	0,45229047	38128,6	44452,5
400	Mean	1,9667E-08	0,00231	1,19333333	6419	6302,3
400	Deviation Standard	5,1316E-09	0,00190874	0,0057735	1873,3	1879,6

Table 5 shows also the Mean numbers of cycles until failure when the temperature is 300ºC and 400ºC. It is noticed that with less temperature the number of cycles until failure is higher. A comparison of the temperatures with respect to the strain rate, shows in low temperature a higher strain rate mean than in high temperature. It is important to mention that the analysis from mathematical point of view cannot be conclusive because there is not clear if with the samples made was obtained a good the representation of the target problem

Table 6 presents a correlation between the quantitative factors of interest. It is not noticed a strong relation between the factors. The factors with more relation are Strain rate with Oxygen, and total strain amplitude withN_f. To this project, it is more important to analyze the factor related to N_f because it explains the number of cycles until failure occurrence.

Table 6: Correlation table

	N_f	C/wt	Strain rate	$\Delta\varepsilon_t$
N_f	1	0,3348415	0,09159558	-0,5425243
C/wt	0,33484147	1	0,59077142	-0,3748172
Strain rate	0,09159558	0,5907714	1	-0,1370734
$\Delta\varepsilon_t$	-0,54252428	-0,3748172	-0,13707343	1

A descriptive analysis is not enough to see the relation between the factors, reason why a multiple linear regression is proposed. Multiple linear regressions are used to explain the relationship between two or more explanatory factors (independent factors) and a response variable (dependent variable) by fitting a linear equation to the observed data.

In this analysis it is assumed that the variable or factor N_f can be explained by the following factors (Table 4):

x_1 = Oxygen; x_2 = Strain rate; x_3=Total strain amplitude; x_4=Temperature.

R project was used because it is more automatic and faster doing the calculations (see Annex I), having obtained the following results:

$$\hat{y} = 1{,}14\text{E}^{+05} + 1{,}069\text{E}^{+09}x_1 - 3{,}873\text{E}^{+06}x_2 - 4{,}16\text{E}^{+04}x_3 - 1{,}22\text{E}^{+02}x_4 \qquad (4.1)$$

The equation 4.1 is an estimation of the regression model, in which:

$\hat{\beta}_0 = 1{,}14\text{E}^{+05}$ (When the parameters are equal to zero, the number of cycles until a failure occurrence grows in units);

$\hat{\beta}_1 = 1{,}069\text{E}^{+09}$ (Per each weight of oxygen, the number of cycles until failure occurrence grows 1,07E+12 units);

$\hat{\beta}_2 = -3{,}873\text{E}^{+06}$ (Per each unit of strain the number of cycles until a failure occurrence decreases 3,87 units);

$\hat{\beta}_4 = -4{,}16\text{E}^{+04}$ (Per each unit of total strain amplitude, the number of cycles until failure decreases in units);

$\hat{\beta}_5 = -1{,}22\text{E}^{+02}$ (Per each level of temperature in 300ºC the number of cycles until the failure decreases units with respect to 400ºC).

Table 8 presents the parameters and the respective coefficient before explained.

To see if all the parameters are relevant in the models, a hypothesis is raised:

$$H_{0i} : \beta i = 0 \ vs \ H_{ai} : \beta i \neq 0 \qquad (4.2)$$

With the Valor-p (see Table 7, column 5^{th}), and with a significance level α=0.05, is concluded that all the parameter are not important for the model. With this conclusion, the model cannot be taken into account for future analysis. The important is to evaluate the possible external factors that influence directly the results.

Table 7: Regression model Parameters

Coefficients:	Estimate Std,	Error	t-value	Pr(>|t|)
(Intercept)	$1{,}14e^{+05}$	2,51E+08	0,454	0,669
X_1 = C/wt	$1{,}069e^{+09}$	8,52E+12	0,126	0,905
X_2=Strain rate	$-3{,}873e^{+06}$	1,27E+10	-0,304	0,773
X_3 =Strain amplitude	$-4{,}16e^{+04}$	3,43E+07	-1.212	0,28
X_4 =Temperature	$-1{,}22e^{+02}$	5,73E+05	-0,214	0,839
Residual standard error:	35370			
Multiple R-squared:	0,3272	Adjusted R-squared:	-0,211	
F-statistic:	0,608	p-value:	0,6749	

4.3 Statistical data analysis for fatigue experiment under corrosion in SCK•CEN

This section has the description and statistical analysis for data collected by experiments SCK•CEN during 2016.

4.3.1 Data description

For the realization of the experiment (see Table 9), the material used was the Annealed stainless steel (see composition Table 8) DEMETRA 316L, with a specimen size 3.2 mm × 7 mm × 70 mm and yield strength around 300 MPa at RT and tensile strength around 600 MPa at RT.

Table 8: Chemical composition

Composition	Cr	Ni	Mo
Wt%	16.82	10.17	2.086

There were performed 5 tests in LBE. The preparing period before making LBE test to reach the low oxygen concentration, were the largest. This conditioning is in terms of corrosion not damaging due to the low temperature and the short exposure time. The specimen was corroded at 2000 hrs in LBE at 500°C.

Table 9: Data base for fatigue experiment

Specimen	**Environment**	$\Delta\varepsilon_t$	$\Delta\varepsilon_e$	$\Delta\varepsilon_p$	**Total stress**	N_f	N_a	$N_f - N_a$	**wppm O2 during test**
2F62	43h, 410-420°C	0,77	0,64	0,13	480,00	18900	19110	210	2x10-11
2F63	36h, 400°C	0,73	0,58	0,15	483,00	16700	16900	200	5x10-12
2F64	70h, 400-405 °C	0,69	0,41	0,28	463,00	69100	69790	690	1,4x10-11
2F67	15h,400°C	0,74	0,47	0,27	505,00	25500	25760	260	7x10-12
2F68	66h,300°C + 20h, 400°C	0,75	0,45	0,30	499,00	32300	32630	330	1,5x10-12
2F65	21h/402 7,5h/420 16,5h/402	0,78	0,51	0,27	478,00	3200	3298	98	8x10-12
2F69	N/A	0,78	0,37	0,41	466,00	13260	12840	420	N/A

The data set (see Table 9) was obtained in SCK•CEN by Marmy Pierre, and in this experiment, the analysis of the corrosion was included.

The specimen 2F65 (see row 6) was corroded for 2000 hrs in LBE at 500°C and tested in LBE. The row 7 (specimen 2F69) was tested in air environment.

4.3.2 Statistical analysis

Preliminary statistical indicators are the mean and the standard deviation (See Table 10). They allow a first approach to the data, in which the mean total stress is 483 units and the Standard deviation is not significant. On the other hand, with regard to the number of cycles until failure, the mean is 32500, and has a standard deviation of 21350 cycles.

Table 10: Descriptive analysis

Trend measures	O2	Total stress	$\Delta\boldsymbol{\varepsilon}_t$	$\boldsymbol{N_f}$	$\boldsymbol{N_a}$
Mean	5x10-12	483	0,736	32500	32838
Standard Deviation	1,8319E-06	16,61325	0,02966479	21349	21552

Table 11 presents a correlation between the quantitative factors of interest in which the factor number of cycle N_f shows a negative correlation with the factors Total stress, $\Delta\boldsymbol{\varepsilon}_t$, $\Delta\boldsymbol{\varepsilon}_e$, and a positive correlation with $\Delta\boldsymbol{\varepsilon}_p$ and $\boldsymbol{N_a}$. With respect to the total strain, it shows also correlation with respect to the factor$\boldsymbol{N_a}$. On the other hand, the factor has a correlation with $\boldsymbol{N_a}$ and $\Delta\boldsymbol{\varepsilon}_e$ with $\boldsymbol{\varepsilon}$.

Table 11: Correlation

	N_f	Total stress	$\Delta\varepsilon_t$	$\Delta\varepsilon_e$	$\Delta\varepsilon_p$	N_a
N_f	1	-0,59799695	-0,8218494	-0,75998627	0,6122719	0,9999999
Total stress	-0,5979969	1	0,5732216	-0,03598673	0,2572305	-0,598294
$\Delta\varepsilon_t$	-0,8218494	0,57322164	1	0,66594966	-0,4321724	-0,8219764
$\Delta\varepsilon_e$	-0,7599863	-0,03598673	0,6659497	1	-0,9605381	-0,7598044
$\Delta\varepsilon_p$	0,6122719	0,25723054	-0,4321724	-0,96053815	1	0,6120047
N_a	0,9999999	-0,59829398	-0,8219764	-0,75980443	0,6120047	1

As Table 11 shows, there are some variable with high levels of correlation between them, reason why factors are going to be excluded. The factors for the regression lineal multiple are:

$$x_1 = \text{Total stress};\ \ x_2 = \Delta\boldsymbol{\varepsilon}_p;\ \ x_3 = \boldsymbol{N_a}$$

In which the response variable is $y = \boldsymbol{N_f}$ number of cycles until the failure.

To calculate the parameter for the regression model R project (see Annexed I) was used. The results are presented in Table 12.

Table 12: Coefficients for the regression model

Coefficients:	**Estimate Std**	**Error**	**t-value**	**Pr(>|t|)**
(Intercept)	$-9{,}39\text{E}^{+05}$	6,07E+05	-1.547	0,365368
Total.stress	$1{,}90\text{E}^{+03}$	1,29E+03	1.471	0,380005
	$-2{,}81\text{E}^{+05}$	2,73E+05	-1.028	0,491178
Na	$9{,}92\text{E}^{+02}$	1,22E+00	815.196	0,000781
Residual standard error	6,178			
Multiple R-squared	1	Adjusted R-squared	1	
F-statistic:	1,592E+07	p-value:	0,0001842	

The results can be given through the equation 4.5);

$$\hat{y} = -9{,}39\text{E}^{+05} + 1{,}90\text{E}^{+03}x_1 - 2{,}81\text{E}^{+05}x_2 + 9{,}92\text{E}^{+02}x_3 \quad (4.5)$$

The interpretation of the model is:

$\hat{\beta}_0 = -9{,}39\text{E}^{+05}$ (when the parameter is zero, each unit of cycles until appear a failure decrease $9{,}39\text{E}^{+05}$ unites);

$\beta_1 = 1{,}90\text{E}^{+03}$ (per each unit of total stress, the number of cycles until appear the failure grow $1{,}90\text{E}^{+03}$unites);

$\beta_2 - 2{,}81\text{E}^{+05}$ (per each unit of elastic strain amplitude, the number of cycles until appear a failure decrease in $2{,}81\text{E}^{+05}$ unites);

$\beta_3 9{,}92\text{E}^{+02}$ (per each unit of microscopic crack amplitude, the number of cycles until the failure growth in $9{,}92\text{E}^{+02}$ unites).

To see if all the parameters are relevant in the models, the following hypotheses are formulated:

$$H_{0i} : \beta i = 0 \ vs \ H_{ai} : \beta i \neq 0 \quad (4.6)$$

With the p-value (see Table 12, column 5th), and with a significance level α=0.05, is concluded the unique parameter relevant for the model is N_a (required to form an already macroscopic crack). It is important to say that as is showed in Table 12, this parameter has a strong correlation with other factors.

To demonstrate the validity of the model it is necessary to confirm these assumptions:

1. The normal distribution of the errors

The normality hypothesis, according with Figure 7, is not verified - the residuals are scattered and not adjusting to the line.

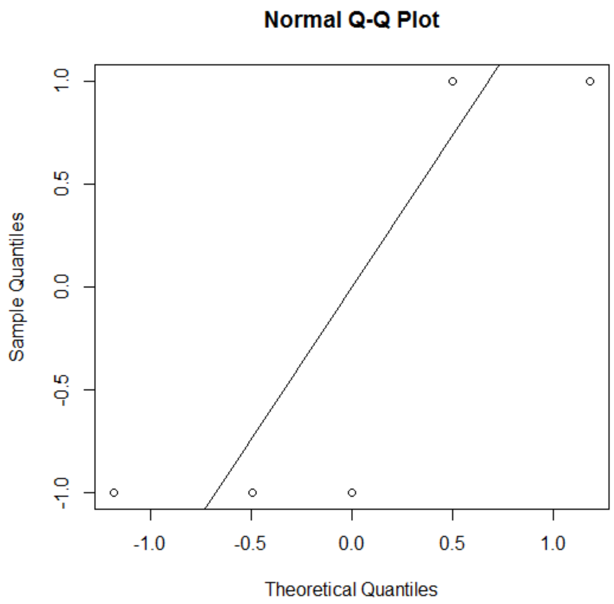

Figure 7: Normality assumption

To demonstrate mathematically this hypothesis (eq. 4.7) the Shapiro test is used.

$$H_0 = \varepsilon \sim N(0,1) \;\; vs \;\; H_1 \neq \varepsilon \sim N(0,1) \qquad (4.7)$$

Assuming α=0,05 and a p-value = 0,00647 from Shapiro-will normality test, what the graphic said is probated, which mean the hypothesis null is rejected, and the model before proposed does not fulfill the normality hypothesis.

2. Assumption of independence

According with Durbin-Watson test, the factors are not independent because the p-value = 2.2E-16 is under α=0,05.

The model is not relevant because two important assumptions are rejected; it is not normal and not independent.

4.4 Synthesis

Observing the statistical result on sections 4.2 and 4.3, in which regression models for the inferential statistic were defined and the results obtained in description statistic with the calculation of the standard deviation, mean and the correlations allow knowing the data behavior. In the case of the regression models not all assumptions were fulfilled. The models then cannot be taken as way to generalize because the analyzed database does not have clear validly. It is the level to which a study establishes the cause-and-effect relationship between the treatment and the observed outcome. Conversely, it refers to the degree to which the absence of a relationship implies the absence of cause; without it, a study is meaningless. In a study that lacks internal validity, the results are probably attributable to a cause other than the treatment. Consequently, the results could not be generalized to similar populations.

The experimental design became an important alternative to start a statistical analysis but true experimental design is one that has at least two independent, parallel groups; randomly assigns subjects to the groups; and assesses treatments prospectively. Studies evaluating experimental research designs have shown that poor execution of specific study procedures can bias results. According with investigations the effects of treatment is 30% greater in studies with inadequate allocation concealment than in studies with adequate concealment. Similar results were observed in studies that lacked appropriate blinding. Less bias was attributed to sequence-generation procedures or to dropouts (Slack & Draugalis, 2001).

The regression was one of the statistical analysis techniques used on the sections 4.1 and 4.2. The unreliable results can occur when: subjects have been selected on the basis of extreme scores, any post-intervention improvement noted could be due partly, if not entirely, to regression rather than to the coping techniques For the data collected, it is not possible to guaranty that any of this situation could not have happened and influenced the final results. An alternative to improve this mistake is clearly preplan the experimental design with all the implication it has and then obtain reliable outcomes that can be statistically tested and based on a mathematical basis.

5. METHODOLOGY PROPOSAL FOR THE ANALYSIS AND DATA COLLECTION

This chapter proposes sample collection planning through experimental design for future projects in order to use analysis techniques that allows extracting relevant information from the data.

5.1 Recognition and statement of the problem

The object important for the study is the vessel of the reactor, and the idea is to propose a methodology for measuring the factors that influence fatigue in crack initiation.

5.1.1 Selection of the dependent and the independent factors

1. Dependent variable

For this project, the response variable is the Number of cycle to rupture (decrease of stress by 20%).

2. Independent factors

According to Argonne National Laboratory (Shack, 2001) there are three important factors that are influencing fatigue crack initiation: Environment, Loading and Material. Those factors studied the reactor on the mechanic fatigue crack initiation, in which it was concluded that the sub-factors with more influence in the crack initiation are: Material variability & experimental scatter, Size effect, surface finish, loading history. Those sub-factors influence the growth of short crack, the nature of the investigation.

Figure 8 shows a cause-effect diagram in which are considered the factors: environment, loading, material, and the “Use method”. The last factor could be consider as a no-controllable sub-factors but is important explain just in as general view because “Use Method” is something that occur implicit to the experiments process.

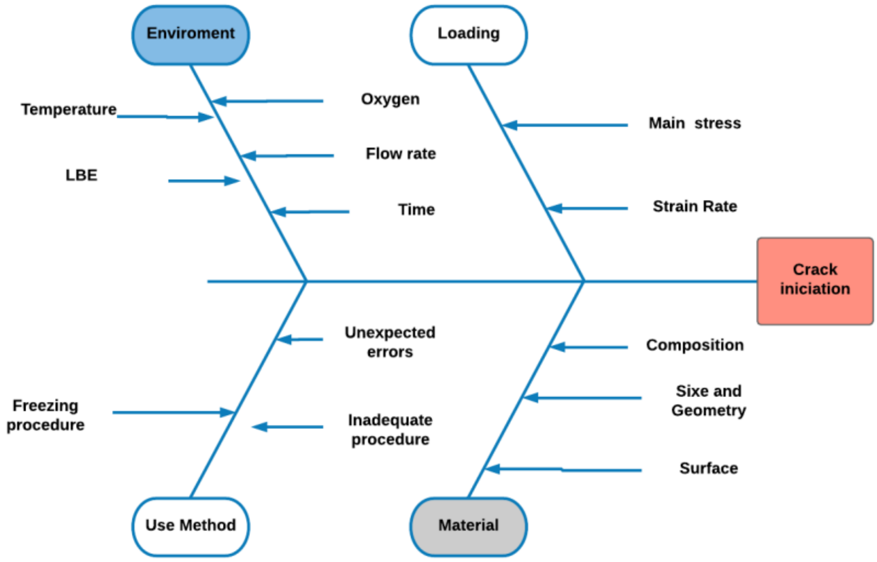

Figure 8: Cause and effect diagram for possible influencing factors in cracking initiation

✓ Material

Describes the factors related to the composition and structures (including defects) of a material that are significant for a preparation, study of properties, (McCauley, 1986).

The sub-factors associated with the factor Material (Figure 8), that could be relevant in the crack initiation are:

1. **Material composition** refers to the science and technology of metals. The subject area can be considered as a combination of chemistry, physics and mechanics with special reference to metals (Seetharaman, 2005).
2. **Size and Geometry**, results from the measurement of geometry and size of a specimen, they are very important and easily quantified.
3. **Processing**, the cold work, heat treatment.
4. **Surface,** in this are implicated the surface finishing and surface preparation.

✓ Loading

Loading represents the external mechanical resistance that a machine acts against. The sub-factors related with, are:

5. **Strain rate**, The Strain is a dimensionless parameter measuring the change in shape of an object relative to its original shape. As such, its strain would be expressed mathematically as:

$$\varepsilon = \frac{L - L_0}{L_0} \qquad (5.1)$$

Where ε is the strain, L_0 is the initial length of the chord and L is the length after deformation . As strain is the change in length (L –L_0) relative to the original length (L_0), it is a dimensionless quantity that is typically expressed as a percentage.

The rate at which the deformation takes place is simply the change in strain (d) per unit of time (dt) and is referred to as the strain rate. (Ruta, J,n.d).

6. **Mean stress**, may be understood as a state of tension experienced by specimen facing extraordinary demands, constraints or opportunities. For this project it represents the average of constant amplitude cycling loading (Rao, 2007).

✓ Environment

The Sub-factors in environment are:

7. **LBE**, Lead Bismuth Eutectic alloy as a coolant in nuclear systems has been investigated for more than half a century. Due to a lack of published experimental data, basic properties such as liquid density, vapor pressure and liquid adiabatic compressibility have been estimated up to the critical point based on empirical and theoretical models by extrapolating the low temperature data of LBE or its constituents (Morita, Maschek, Flad, Yamano, & Tobita, 2006).
8. **Temperature**, the temperature is the measure of the hot or cold scale of an object through a thermometer; the result can be given in different units of measurement: Celsius, kelvin and Fahrenheit.
9. **Oxygen**, the oxygen is a chemical element that is a gas without smell or color. Oxygen forms part of the air on earth.
10. **Flow rate**, the flow rate is calculated to determine at what rate, usually stated in drops per a defined time, an element (water, LBE) flow, which means the speed of the coolant.

11. **Time**, Time is a dimension and measure in which events can be ordered from the past through the present into the future, and the measure of durations of events and the intervals between them (Demetris, 2013). In this project is the exposition time.

- ✓ Use method

The factor “use method” is relevant due to the many unexpected error always present in a research, in this case is evaluated:

12. **Inadequate procedure**, Error because of procedure in any process, like for example freezing procedure.
13. **Unexpected errors**, random mistakes in the process.

5.2 Choice of factors, levels, and ranges

The factors before mentioned must be classified into three groups, the held-constant, controllable and noise factors. As SCK•CEN has been developing research about the fatigue under LBE, for this section is taken into account the results and the procedures that have been done before, with the idea to add the necessary to make general results.

5.2.1 Held-constant factors

In the group of the Held-constant is the factor Environment with the sub-factor LBE, it is constant, because it is the environment selected for this study due to the few research developments in the area with these characteristics.

Other factor part of this group is Material, with the sub-factors size and geometry, composition and dimension. The material is going to be the same for every specimen, for LBE has been found a compatibility with 316L steel, reason why this is the material that is going to be used (Marmy & Gong 2015).

5.2.2 Controllable factors

In this group, the factor Environment is considered a controllable with the sub-factors: Temperature, oxygen and flow rate. The first two sub-factors have been recognized with great importance due to Its possible effect in the fatigue crack initiation.

- ✓ According to Energy Nuclear Angengy (2015) the temperature, in LBE operate normally between 500±50 °C, where 450°C is a moderate level of temperature and 550°C is a high level.

- ✓ The oxygen concentration in LBE affects the corrosion mechanism of steels. At low oxygen concentration the steel exhibit weight loss, and the corrosion is due to the dissolution of alloy elements. At high oxygen concentration exhibit weight gain, and the formation of protective thin oxide layer on the surface of specimens has been detected (Aiello et al., 2004). Considering the results mentioned before, the behavior at low and high oxygen should be analyzed and compared.
- ✓ According to Agostini et al. (2005), the LBE and flow rates, covering the nominal range from 50 to 350 kg/s, is showed through the characteristic curve that is satisfactory, the test was made with experimental data at flow rates higher than 100 kg/s; at flow rates, lower than 100 kg/s .

The factor Loading also belongs to this group with two sub-factors the strain rate, and the mean stress.

- ✓ According to (Marmy, 2012) the occurrence of Dynamic strain ageing (DSA) makes 316L steel sensitive to strain rate and strongly affects its fatigue endurance. In the papers, it is common to see strain levels between $(6.67x10^{-8}ms^{-1}\ to\ 6.67x10^{-3}ms^{-1})\ and\ (1x10^{-3}ms^{-1}\ to\ 1x10^{-6}ms^{-1})$. Observing the pattern in other investigations, the suggestion could be high strain 10^{-3} and low strain10^{-6}.
- ✓ The experiment realized by (Strizak, 2003), used high mean stress conditions (R=0.3, 0.5, and 0.75). To cover every situation, it would be better to use a low and high mean stress and see the behavior.

For the sub-factors in general, it is suggested a level but each of them may change according to the investigator and the objective set.

5.2.3 Uncontrollable factors

There are some noise factors, such as: Error freezing, inadequate procedure, unexpected errors, metallurgy in the relation to grain in the initial material, Surface, that can be difficult to measure.

5.3 Experimental design (Taguchi Method)

The most important factors to analyze according with the literature review, and self-invention throe the people with experience in the field are: Environment with the sub-factors temperature, flow rate and oxygen, loading with strain rate.

Table 13: Factors for the experimental design

Factors	**Sub-factors**	✓ **Levels**	**+ Level**
Loading	Strain rate (S)	10^{-3}	10^{-6}
Environment	Temperature (T)	550°C	450°C
	Oxygen (O)	high oxygen	low oxygen
	Flow rate (F)	>100 kg/s	<100 kg/s

After choosing the important factors and sub-factors and without a strong background with the specific characteristic of this project, it is necessary to select the most important interactions, to reduce cost and time in future experiments. A strategy for it was to analyze literature and determine if the possible interaction is relevant, (See Table 7). The possible interactions are:

- ✓ The interaction between the sub-factors **Strain rate and Temperature** was analyzed by Van Den Bosch, Sapundjiev, & Almazouzi (2006). The strain rate on T91 material was tested in LBE environment. The objective of the experiments was to study whether the susceptibility to liquid metal embrittlement (LME) or the absence of LME is influenced by the temperature or the applied strain. Generally, when solid metals are exposed to liquid metals and stress is applied, they may undergo abrupt brittle failure known as liquid metal embrittlement. The liquid metal embrittlement has been investigated in the temperature interval 150–450 ºC, at strain rates between $1x10^{-3}s^{-1}$ and $1x10^{-6}s^{-1}$. The results showed that varying the strain rate does not have an influence on the susceptibility to LME of the untreated T91 material. At temperatures above 300 ºC the total elongation of the untreated T91 is greater in liquid metal than that in gas environment when comparing to reference data from the same batch of T91. The results of the experiment are not clear with respect to this specific project but shows that the interaction is possible (Van Den Bosch, Sapundjiev, & Almazouzi, 2006).

- ✓ **Strain rate and Oxygen.** According to Van der Piascik *et al*(1997), the available data on the effect of various materials and loading factors such a, steel type, dissolved oxygen level, strain rate and sulfur content of the fatigue life of carbon steel. Considering that have been studied before, it is interesting to analyze it.
- ✓ **Strain rate and Flow rate**. The high strain rate dependence of the flow stress of metals and alloys is described from a dislocation mechanics viewpoint over a range beginning from conventional tension/compression testing through split Hopkinson pressure bar (Armstrong & Walley, n.d.).
- ✓ **Temperature and Oxygen**. (Niu, Tian, Zhang, Gao, & Wu, 2014) explain the corrosion in LBE through the experiments that were conducted with three different oxygen concentrations of $4.6 \times 10 - 7wt\%$, $1.63 \times 10 - 5wt\%$, and $5.84 \times 10 - 5wt\%$, typically representing the low, medium and high oxygen content in LBE within the temperature range 395–520 °C. The results show that the fouling factor increases significantly with the oxygen concentration in the range of 1.0×10-5wt%, because the oxide layers begin to form in this range of oxygen concentration (Niu, Tian, Zhang, Gao, & Wu, 2014). The conclusion in the paper makes believe that this interaction can be important for this project.
- ✓ **Temperature and Flow rate**. For this study were selected three steels of material as the mold substrate materials in plastic injection forming. The plasma immersion ion implantation (PIII) system with an electron cyclotron resonance microwave source was applied to prepare the specimens by varying the implantation temperature (400 °C, 460 °C and 520 °C) and the volume flow rate ratio (4:1, 1:1 and 1:3) of nitrogen to hydrogen in the gas mixture (N2:H2). For the specimens with the same substrate material, the mean hardness was either invariant to or lowered by increase in the total penetration depth of nitrogen. The conclusion showed that the total penetration depth of nitrogen in a specimen is increased by elevating the implantation temperature, irrespective of the substrate material. A clear and definite relationship cannot be found between the implantation temperature and the peak position of nitrogen concentration. The effect of the N2:H2 ratio on either the total penetration depth or the peak position of nitrogen cannot be defined clearly too (Jen, 2007). This paper does not give a conclusion answer for the approaches in this investigation but set a point that the relations between these sub-factors have been already studied and could be important to analyze for the study of the crack initiation.

- ✓ **Oxygen and Flow rate**. (Yuan, 2011) Made the study of the Influence of Oxygen Flow Rate on the Variation of Surface Roughness of Fused Silica during Plasma Polishing Process, for the development of it. The plasma was generated by using 13.56 MHz and 100 MHz RF power source respectively. Pre-polished fused silica substrates with average surface roughness of 1.5nm (rms) were used for the investigation of the plasma polishing process the gas flow of SF6 was fixed at 10 SCCM, the total flow rate of the carrying gases was selected as 110 SCCM. The results showed that In the CCHC RF plasma polishing processes, the variation of the surface roughness of the fused silica was influenced by the discharge frequency, the carrying gas, and the gas flow rate ratio of the carrying gases (Yuan, 2011) . According to this paper, it is possible to find an interesting relation between these parameters, reason the interaction of these factors would be considered (see Table 14).

Table 14: Interaction of the experimental design

Numbers	Sub-factors interaction		Relevance
1	Strain rate and Temperature	SxT	Important
2	Strain rate and Oxygen	SxO	Important
3	Strain rate and Flow rate	SxF	Important
4	Temperature and Oxygen	TxO	Important
5	Temperature and Flow rate	TxF	Important
6	Oxygen and Flow rate	OxF	Important

After a theoretical evaluation of the important parameters, with four factors of 2 levels and six interactions, the orthogonal Taguchi to be used would be the L16 matrix, which means that it will execute 16 tests or experimental conditions (See Table 15).

Table 15: Taguchi arragment

Run	S	T	SxT	O	SxO	TxO	e1	F	SxF	TxF	e2	OxF	e4	e5	e6
Colum	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	1	1	1	1	2	2	2	2	2	2	2	2
3	1	1	1	2	2	2	2	1	1	1	1	2	2	2	2
4	1	1	1	2	2	2	2	2	2	2	2	1	1	1	1
5	1	2	2	1	1	2	2	1	1	2	2	1	1	2	2
6	1	2	2	1	1	2	2	2	2	1	1	2	2	1	1
7	1	2	2	2	2	1	1	1	1	2	2	2	2	1	1
8	1	2	2	2	2	1	1	2	2	1	1	1	1	2	2
9	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
10	2	1	2	1	2	1	2	2	1	2	1	2	1	2	1
11	2	1	2	2	1	2	1	1	2	1	2	2	1	2	1
12	2	1	2	2	1	2	1	2	1	2	1	1	2	1	2
13	2	2	1	1	2	2	1	1	2	2	1	1	2	2	1
14	2	2	1	1	2	2	1	2	1	1	2	2	1	1	2
15	2	2	1	2	1	1	2	1	2	2	1	2	1	1	2
16	2	2	1	2	1	1	2	2	1	1	2	1	2	2	1

Table 15 is built through the Figure 9, in which the figure at the Right represent the letters and the figure on the left are the location. For this case A= S, B= T, D= O and H=F. Thus, S is in the position 1, T is in the position 2, ST is in the 3th position, O is at the position 4, OS is the 5th position and OT the 6th position, F is at position 8, FS is in the position 9, FT at the position 10 and FO at the position 12.

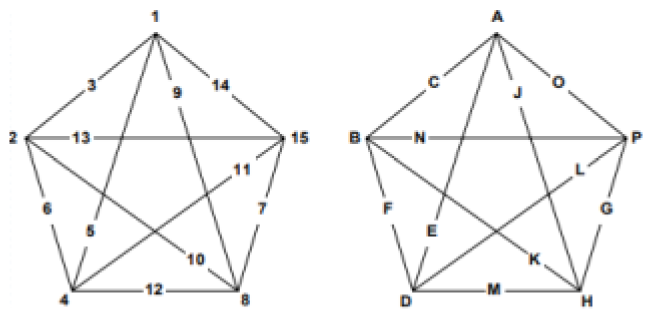

Figure 9: Lineal graph for L16 array

5.4 Sample size

The Taguchi technique that for this dissertation seeks to reduce cracks initiation in the vessel allows designing the experiment which will involve 16 runs.

To find the combination of factors levels that maximize the number of cycles until crack initiation, each combination proposed by the orthogonal array should be replicated, considering noise factors. The noise factors associated with this process are the people who perform the assays and the type of experimental machine (See Figure 7). Due to the costs associated with the replication, for this project, at least one replica of each assay is suggested, that would consist of 2*16 runs, which means in total 16 samples with 2 replications each.

6. Conclusion

The information over nuclear environment and crack initiation has been extensively studied in different environments, as was shown through the background studies in Chapter 2, and in the review made in chapter 3. But in many of the cases evaluated in this dissertation, it was common to see the absence of specialized statistical techniques as a tool but also the design of data collection for the development and processing of the information.

In chapter 4, two set of data were analyzed based on some statistical techniques as regression model, and indicators. For analyzed data sets, it was not possible to propose a model that comply with the assumptions, but in this case, it is important to emphasize that the databases lacked a solid statistical base of sample design. There was no knowledge of the errors assumed at the time of data collection. So, this information was limited to that fact.

Since the models proposed in Chapter 4 are not valid because there was no proper sample design, in chapter 5 a methodology to experiment planning is proposed. Experimental design proposes guidelines to carry out a proper experiment. To select the factors to be part of the experiment, It was not possible to evaluate the relevance of the parameter in a mathematical way, reason why literature was used to make a review about the topics and with it, evaluate the relevance of the possible parameters. The levels for the parameters were also suggested but it is necessary to underline that, those levels could be modified according with the necessity and the objectives of the investigator. On the other hand, a sample size of 32 was determinate.

The project set a base for a futures research in order to follow a proper mechanism of analysis that will help to get mathematically reliable results.

6.1 Discussion

When using the Taguchi method and not others it is always with the objective of working with the factors that are really important and thus reducing the size of samples, in this case due to the size of the matrix this was not directly reflected but it is still a valid form for the solution to the problem presented in this project and it is open the possibility of using any other technique of experiment design according to the needs of the researcher.

On the other hand, the orthogonal matrix (see Table 16) leaves some columns free for the inclusion of factors and interactions, it is worth noting that a proposal is made in this project with regard to the factors subject to changes based on the needs of the project

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ANNEX I – R PROJECT CODES AND PACKAGES

```
# Codes for the model adjusted #

datosWeb3 <- read.delim('clipboard', dec=",") # This function is to call the data

attach(datosWeb3) # Atached the datas

base=data.frame(N_f,C.wt,Strain.rate,X.._t) # The factors parenthesis became a new table

cor(base) # Calculate the correlation coefficient for the table "base"


library(lmtest) # Package necessary for the model

ajuste <- lm(N_f ~ C.wt + Strain.rate + X.._t+Test.Temperature)  # Stimation of the model

summary(ajuste) # Sumarry of the model
```