CHARPY IMPACT AND TENSION TESTS OF TWO PIPELINE MATERIALS AT ROOM AND CRYOGENIC TEMPERATURES

A THESIS SUBMITTED TO THE GRADUATE SCHOOL OF APPLIED SCIENCES OF NEAR EAST UNIVERSITY

By
Abdulkarim Omar Alfitouri

In Partial Fulfillment of the Requirements for the Degree of Master of Science in Mechanical Engineering

NICOSIA, 2018

Abdulkarim Omar Alfitouri
CHARPY IMPACT AND TENSION TESTS OF TWO PIPELINE MATERIALS AT ROOM AND CRYOGENIC TEMPERATURES
NEU 2018

CHARPY IMPACT AND TENSION TESTS OF TWO PIPELINE MATERIALS AT ROOM AND CRYOGENIC TEMPERATURES

A THESIS SUBMITTED TO THE GRADUATE

SCHOOL OF APPLIED SCIENCE

OF

NEAR EAST UNIVERSITY

By

Abdulkarim Omar Alfitouri

In Partial Fulfillment of the Requirements for
the Degree of Master of Science
in
Mechanical Engineering

NICOSIA, 2018

Abdulkarim Omar Alfitouri: CHARPY IMPACT AND TENSION TESTS OF TWO PIPELINE MATERIALS AT ROOM AND CRYOGENIC TEMPERATURES

Approval of Director of Graduate School of

Applied Sciences

Prof. Dr. Nadire ÇAVUŞ

We certify this thesis is satisfactory for the award of the degree of Master in Mechanical Engineering

Examining Committee in Charge:

Prof. Dr. Yusuf ŞAHİN	Committee Chairman, Mechanical Engineering Department, NEU
Assoc. Prof. Dr. Kamil DİMİLİLER	Automotive Engineering Department, NEU
Dr. Ali Şefik	Mechanical Engineering Department, NEU
Assist. Prof. Dr. Ali EVCİL	Co-Supervisor, Mechanical Engineering Department, NEU
Prof. Dr. Mahmut Ahsen SAVAŞ	Supervisor, Mechanical Engineering Department, NEU

I hereby declare that, all the information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Name, Last Name :

Signature :

Date:

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude and thanks to my supervisors Prof. Dr. Mahmut A. SAVAŞ and Assist. Prof. Dr. Ali EVCIL for their guidance, suggestions and many good advices and their patience during the correction of the manuscript. They have been my mentor and my support at all the times. I am very thankful to them for giving me an opportunity to work on interesting projects and for their constant encouragement and faith in me. I am immensely grateful for your kindness, patience, time and professional contributions to the success of my study. Thanks for always pushing me for more.

I could not carry out this research without the support and help of Libyan Iron and Steel company and Misurata factory for plastic pipes. I am gratefully to them

I would also like to express heartiest thanks to my parents, my wife, Amna, and my family members, Omar, Abdulrahman, Fatma, Ali, Abdumalik, Abdurlbari and Eshtewi, for their patience, ever constant encouragement and love during my studies.

To my parents ...

ABSTRACT

There are a number of material alternatives for pipelines used to transport oil, natural gas and water for long distances. For instance, the majority of pipelines in Libya are made of API 5L X60 steel. The water is transported across the Mediterranean sea from Turkey to Cyprus island by means of a 80 km long HDPE (High Density Polythylene) pipeline. In the present study, the tensile, Rockwell hardness and Charpy impact tests of the oil pipeline steel API 5L X60 were carried out both at RT (Room Temperature) and also at NT (Liquid Nitrogen Temperature) following either ASTM or EN ISO standards. The same procedure was also followed to characterize the same properties of the HDPE samples. It was found that the Charpy impact properties of the notched API 5L X60 steel samples were reduced drastically from 210J to 5J once cooled down to liquid nitrogen temperature. Nevertheless, the tensile strength at room and liquid Nitrogen temperatures were on average 498MPa and 580 MPa, respectively. The Rockwell Hardness B Scale was found as 65 at room temperature and 88 when cooled in liquid nitrogen. Both the tensile strength and also fracture elongation of the HDPE were reduced when tested at liquid nitrogen temperature. Its tensile strength was found as 470 kPa at RT whereas it dropped to 130kPa at liquid nitrogen temperature. Its fracture elongation was also reduced from 368 % to 65 % when cooled down to liquid nitrogen temperature. The Charpy impact energy of the HDPE was dropped from 122 kJ/m^2 to 44 kJ/m^2 when cooled down to liquid nitrogen temperature. The examination of fractured samples showed that the un-notched API 5L X60 steel samples did not lose their ductile fracture behavior when cooled down to liquid nitrogen temperature. However, this was not observed in the HDPE samples. Thus, HDPE did not appear to be a suitable material for sub-zero temperature use.

Keyword: API 5L X60 Steel; HDPE; Charpy impact test; tensile test; Rockwell hardness

ÖZET

Petrol, doğal gaz, su gibi hayati önemi olan akışkanları uzun mesafelere taşıyan boru hatları farklı malzemelerden imal edilebilir. Örneğin, Libya'daki petrol boru hatları genellikle API 5L X60 çeliğinden yapılmıştır. TC'den KKTC'ye tatlı su nakli Akdeniz'de iki kıyı arasında uzanan 80 km'lik HDPE (Yüksek Yoğunluklu Polietilen) boru hattı ile sağlanmıştır. Bu çalışmada, API 5L X60 çeliğinden hazırlanan numunelere oda sıcaklığında çekme ve Charpy darbe testleri uygulanmış, ayrıca Rockwell sertlik değerleri ölçülmüştür. Deneyler farklı sürelerde sıvı azot içerisinde soğutulmuş numuneler için de tekrarlanmıştır. Bir karşılaştırma yapmak üzere, benzer çalışmalar HDPE numuneleri üzerinde de gerçekleştirilmiştir. Çentikli API 5L X60 çeliği numunelerinde Charpy darbe testi değerlerinin oda sıcaklığında 210 J iken sıvı azotta soğutulmuş numunelerde 5 J'a kadar düştüğü gözlenmiştir. Buna karşın, çentiksiz numunelerde Charpy darbe testi değerlerinde bir azalma izlenmemiştir. Benzer biçimde, sıvı azot içerisinde soğutulmuş çekme testi numunelerinin çekme mukavemetlerinde de düşme görülmemiş; önemli bir miktar yükselme izlenmiştir. Oda sıcaklığında, Rockwell B ölçeğinde 65 olan sertlik değeri sıvı azot içerisinde soğutulduğunda 88 olarak tesbit edilmiştir. Diğer yanda, HDPE numunelerde oda sıcaklığında 470 kPa olan çekme mukavemetinin sıvı azotta soğutulmuş numunelerde süreye bağlı olarak 130 kPa değerine indiği tesbit edilmiştir. Kopma uzaması da %368'den %65 seviyesine inmiştir. Oda sıcaklığında yapılan Charpy darbe testinde çentiksiz numunelerde 122 kJ/m^2; çentikti numunelerde ise 44 kJ/m^2 değerleri elde edilmiştir. Test numunelerinin kırılma bölgeleri incelendiğinde çentiksiz API 5L X60 çelik numunelerin oda sıcaklığında olduğu gibi, sıvı azot sıcaklığına soğutulduklarında da sünek biçimde davrandıkları gözlenmiştir. Çentiksiz HDPE numuneleri ise soğutulduklarında süneklikleri kaybetmiş ve gevrek bir kırılma davranışı göstermişlerdir. HDPE'nin düşük sıcaklıklarda kullanıma uygun bir malzeme olmadığı anlaşılmaktadır.

Anahtar Kelimeler: APL 5L X60; HDPE; Charpy darbe testi; çekme testi; Rockwell sertlik değeri

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

$\boldsymbol{a}$	Height
$\boldsymbol{A_o}$	Cross-sectional area
$\boldsymbol{E}$	Young's modulus
$\boldsymbol{E_i}$	Initial energy
$\boldsymbol{E_k}$	Kinetic energy of the pendulum
$\boldsymbol{E_r}$	Energy after the rupture
$\boldsymbol{E_p}$	Potential energy
$\boldsymbol{F}$	Force
$\boldsymbol{g}$	Gravity
$\boldsymbol{l}$	Length of the rod after loading
$\boldsymbol{l_0}$	Original length of the rod
$\boldsymbol{m}$	mass of the hammer
$\boldsymbol{v}$	Speed of pendulum
$\boldsymbol{\nu}$	Poisson's ratio
$\boldsymbol{\sigma_{eng}}$	Engineering stress
$\boldsymbol{\sigma_T}$	True stress
$\boldsymbol{\varepsilon_{eng}}$	Engineering stain
$\boldsymbol{\varepsilon_T}$	True strain

CHAPTER 1
INTRODUCTION

1.1 Research Background

The pipeline is a convenient, economic and safe way of transporting petroleum, natural gas and water with high pressure and speed for long distances (Thomas & Dawe, 2003). In recent years, the demand for petroleum and natural gas is gradually on the increase, and the capacity of oil– gas pipeline transportation has been developing greatly. Transmission pipelines have a good safety record due to a combination of good design, materials and operating practices (Macdonald & Cosham, 2005). However, like any engineering structure, the best-designed and maintained pipeline may become defective as it progresses through its design life.

The old pipes laying in the ground are made from the full spectrum of materials, such as cast and ductile irons, asbestos cement, steel, PVC and PE.

Steel is arguably the world's most "advanced" material. It is a very versatile material with a wide range of attractive properties which can be produced at a very competitive production cost (Sinha, 1989; Bello, 2007). The complexity of steel arises with the introduction of further alloying elements into the iron-carbon alloy system (Keehan, 2004). The optimization of alloying content in the iron carbon alloy system, combined with different mechanical and heat treatments lead to immense opportunities for parameter variations and these are continuously being developed. Pipeline steels have for many decades been in demand but are becoming vital because there is an expansion in the need to transport liquid as gas fossil fuels over large distances and in dire environments. There are many essential properties for pipeline steels.

High density polyethylene (HDPE) is also used as a drainage pipe material because it is lightweight, corrosion resistant, easy to install, and has a low maintenance cost (Hsuan, 1999). The design of HDPE corrugated drainage pipe is based on the assumption that the pipe will deform and thus relieve stress (Hsuan, 1999). HDPE has become the leading polymeric material for gas and water pipelines due to its many advantageous properties over metal such as lower weight, higher chemical and corrosion resistance, ease of bonding and low delivery, construction and maintenance costs.

Two basic types of impact testing have evolved: (1) Bending which includes Charpy and Izod tests, and (2) tension impact tests (Singh, 2009). Bending tests are most common and they use notched specimens that are supported as beams. In the Charpy impact test, the specimen is supported as a simple beam with the load applied at the center (Mechanical Engineering, 2016). In the Izod test, the specimen is supported as a cantilever beam (Mechanical Engineering, 2016). Using notched specimens the specimen is fractured at the notch (Mechanical Engineering, 2016). Stress is concentrated and even soft materials fail as brittle fractures. Bending tests allow the ranking of various materials and their resistance to impact loading. Additionally, temperature may be varied to evaluate impact fracture resistance as a function of temperature. Both Charpy and Izod impact testing utilize a swinging pendulum to apply the load (Murray et al., 2008). On the other side, the tensile impact test avoids many of the pitfalls of the notched Charpy and Izod bending tests. The behavior of ductile materials can be studied without the use of notched specimens. Pendulum, drop-weights and flywheels can be used to apply the tensile impact load. The notched bar tests are extensively used of all types of impact tests Therefore, the impact measures the energy necessary to fracture a standard notched bar (i.e. notch toughness) applying an impulse load or sudden load (Singh, 2009). The notch provided on the tension side in the specimen locates the point of fracture (i.e. acts as stress concentration point). All forms of the impact test depend upon the swinging pendulum (Singh, 2009). The height from which it drops is a measure of its inertia at the lowest point. There it collides with the specimen, breaking latter and continuing onward in its swing. The height to which the Pendulum rises is dependent upon the inertia left in the pendulum after breaking the specimen (Singh, 2009). The difference between height and the height to which it would have risen, had no specimen been present is a measure, the energy required to break the specimen. This, expressed in Joules (i.e. N-m), is the impact value of the specimen. A high impact value indicates better ability to withstand shock than an impact value (Singh, 2009).

Engineers use metallic materials in designing structures and machine elements which are almost always subject to external loadings and environmental conditions. Metallic materials fail in different modes depending on the type of loading (tensile, compressive, bending, shearing, or torsion) and on the service conditions (temperature and corrosivity of the environment) (Matsagar, 2015). Strength is of little use without toughness and there is usually a trade-off between the two. Toughness is generally expressed as impact toughness since in the majority of circumstances it is measured using a Charpy or Izod impact notch test (Lucon, 2015). Ductility is a measure of the degree of plastic deformation that the metal can sustain before fracture (Keehan, 2004). It is important for a designer to know how much plastic deformation will be experienced before fracture in order to avoid disastrous consequences in certain applications (Keehan, 2004). It may be measured by percentage elongation or area reduction of tensile specimens (Keehan, 2004).

1.2 Objectives of the Project

In this work, there are several objectives on studying mechanical testing on API 5L X60 and High Density Polyethylene (HDPE) pipeline. The API 5L X60 is a commonly used in pipeline steel in Libya. The HDPE polyenes is used in the pipeline that transports water across Mediterranean sea from Anamur (Turkey) to Gecitkale (TRNC). Two main objectives that are needed to be achieved at the end of this study are:

- To understand the changes in mechanical behavior of API 5L X60 steel and HDPE polymer as a result of impact test and tensile tests.
- To understand the effect of liquid Nitrogen temperature treatment on the mechanical behaviors with composition variations in the API 5L X60 steel and HDPE samples.

1.3 Thesis layout

The thesis is arranged as follows:

Chapter 2; Literature Review: Critical literature review focusing on the properties of materials and the effects of inclusions in polymers. Also, the chapter includes theoretical background relevant to the mechanical tests of the material.

Chapter 3; Methodology: sample preparation and test procedures are provided.

Preliminary test results are presented to show the quality of the data and highlights the issues related to sample production.

Chapter 4; Results and Discussion: Results and discussion of the impact of various parameters of the pipeline steel samples and HDPE samples in different stress modes such as tension and Charpy impacts are given.

Chapter 5; Conclusions and Recommendations: The findings are summarized, the conclusions are derived, the shortcomings of the current research are noted and the directions of further possible research are proposed.

CHAPTER 2
LITERATURE REVIEW

2.1 Pipeline Materials

2.1.1 Polymeric Materials

The word polymer is derived from Greek, poly means many and meros meaning parts (Katz, 1998). A polymer consists of very large molecules made up of many smaller units called monomers which are joined together to form a long chain by the process of polymerization. Monomers are called the building blocks of polymers; monomers constitute mostly hydrogen and carbon. Sometime oxygen, nitrogen, chlorine, or fluorine is added to monomers to create different properties and grades of polymers (Farshad, 2006).

Polymers such as latex from trees, protein from animals and silk from silk worm, are a few examples of naturally occurring polymers, which are appropriately called natural polymers (Katz, 1998). Polymers, other than natural polymers, are called synthetic polymers, which are manmade polymers, e.g., Bakelite, polyethylene, epoxy, PVC, silicone etc. Synthetic polymers are further divided into three categories thermosetting plastics, thermoplastic, and elastomer (PPFA, 2005; Katz, 1998). Thermoplastic plastic refers to a plastic that can be repeatedly softened by heating and hardened by cooling through a temperature range characteristic of the plastic, and that in the softened state can be shaped by flow into an article by molding or extrusion (PPFA, 2005). Thermosetting Plastic refers to a plastic that, when cured by application of heat or by chemical means, changes into a substantially infusible product (PPFA, 2005).

The discovery of polyethylene accidentally occurred during 1894 when an experiment by Hans von Peckmann yielded decomposition of diazomethane in the form of white powder. Further analysis indicated that the product was made up of hydrogen and carbon atoms forming a long chain of methylene (CH2) molecules which are known as polymethelenes. The second attempt to create polyethylene was made in 1929 by Fredrick and Marvel, who were successful in producing a polyethylene with lower molecular weight by heating butyllithium (BuLi) (Storm and Rasmussen, 2011). In 1933, two English researchers at Imperial Chemical Industries (ICI) in England, namely Eric Fawcett and Reginald Gibson, were conducting an experiment on 16 an ethylene and benzaldehyde mixture at very high temperatures when a sudden loss of pressure in the experimenting vessel resulted in a waxy solid, which they called polyethylene. It was the first polymerization of the ethylene monomer. ICI began the commercial production of polyethylene in 1939. DuPont was the first industry to manufacture low density polyethylene (LDPE) collaborating with ICI to produce the first LDPE product for the U.S. Government in 1943 (Storm and Rasmussen, 2011).

According to Hsieh et al. (2007), the plastic pipe system was introduced during 1930s and it was accepted globally during late 1950s and early 1960s. The confidence of usage of HDPE pipe in underground infrastructure has increased during every decade since the 1970s (Kuffer and Freed, 2009). Similarly, Watkins (2004) states the usage of HDPE pipe in underground infrastructure is significantly increasing due to its unique 18 properties such as its light weight and resistance to corrosion and abrasion as well as the fact that it is easily molded, extruded, machined and welded. “PE is the most widely used polymer in the world, and PE water pipes are increasingly being installed in buried and building plumbing applications globally” (Welton et al., 2010). “PE pressure pipes have excellent records of performance only some abnormal service loadings may result in field failures” (Yayla and Bilgin, 2006). HDPE pipes are used to carry potable water (Whelton et al., 2011; Zhao et al., 2002), and the use of PE to supply drinking water has been increasing in the Danish market since 1960 (Denberg, 2009). PE has been successfully used primarily in water utilities and in the gas industry for over 50 years (Allwood and Beech, 1993, Haager et al 2006). There are several factors that have influenced the usage of HDPE pipe for water distribution. These include flexibility, cost of installation and manufacturing, resistance to oxidants, corrosion, and abrasion, long-term performance, low thermal conductivity and squeeze-off properties, Squeeze-off is the emergency situation to stop or nearly stop the flow in PE by flattening the pipe between parallel bars. This is method is used when carrying repair or maintenance work of PE (Yayla and Bilgin, 2006), (Watkins, 2004; Welton et al., 2010; Yayla and Bilgin, 2006; Denberg, 2009; Frank et al., 2009).

2.1.1.1 HDPE

According to Plastic Pipe Institute (2008) and Watkins (2004),“High Density Polyethylene (HDPE) was first invented in England in 1933 by Imperial Chemical Company (ICI). The early polymerization processes used high-pressure (14,000 to 44,000 psi) autoclave reactors and temperatures of 200°F to 600 °F. It was produced in a free radical chain reaction by combining ethylene gas under high pressure with peroxide or a trace amount of oxygen. Later in the 1950’s, polyethylene(PE) with low pressure was introduced. Polyethylene as a density varying between 0.935 to 0.941 g/cm^2 (58.37 to 58.74 pcf) for medium density polyethylene, and 0.941 to 0.945 g/cm^2 (58.74 to 58.99 pcf) for high density polyethylene. Industry practice has shown that base resin densities are in the range of 0.936 to 0.945 g/cm^2 (58.43 to 58.99 pcf). The polyethylene pipes with higher density, such as0.952 g/cc(59.43 pcf),in combination with higher molecular weight and bimodal molecular weight distribution recognized higher levels of performance under ISO (International Organization for Standardization) standards for PE piping outside North America).”According to AWWA (2006), “Polyethylene (PE) is a semi crystalline polymer composed of long, chain-like molecules of varying lengths and numbers of side branches.” The above definition describes the structure of the polymer, which means that many parts are joined together (cross-linked) to make a whole. The structure of high density polyethylene is stronger when compared to two types of PE (i.e., LDPE & MDPE); the molecular weight is the main factor that determines the durability. The long-term strength, toughness, ductility and fatigue endurance improve as molecular weight increases. Also, the amount of crystallinity is 65% in high density polyethylene (HDPE) compared to medium density. As HDPE’s crystallinity increases, its stiffness, modulus, and chemical resistance increases, while its permeability, elongation at failure, and flexibility decreases (Koerner, 2012). The mechanical properties of HDPE pipe material seen in Table 2.1 taken from reference (ISCO Industries, 2000)

Table 2.1: Typical physical properties of HDPE (ISCO Industries, 2000)

Property	Specification	Unit	Nominal value
Density	ASTM D 1505	Gm/cm^3	0.955
Hardness	ASTM D 2240	Shore "D"	65
Compressive strength (yiled)	ASTM D-695	Psi	1600
Elongation @ yield	ASTM D-638 (2"/min)	Psi	3200
Elongation @ break	ASTM D-638	%, minimum	750
Impact Strength (IZOD)	ASTM D-256	In-lb/in notch	42

2.1.2 Steels

Carbon steel is one of the most common types of steel used in many different industries such as construction industry, fabrication of pipelines, and many other applications. As carbon content increases in the steel, hardness also increases and hardenability is enhanced. But the problem with high content of carbon is that the steel becomes more brittle and the weldability decreases. In most cases, the steel will have to be welded together. For that reason the carbon content must be chosen according to the requirements of the application. There are several elements that could be added to carbon steel in order to improve certain mechanical or physical properties. Such elements could be manganese, phosphorus, sulphur, and silicon. These elements are called alloying elements. However, in plain carbon steels, the main characteristics and the weldability depend on the carbon content. The plain carbon steels are then further divided into low carbon steels, medium carbon steels, high carbon steels, and very high carbon steels (Capudean, B. 2003).

Also called mild steels, as carbon content could only reach up to maximum 0.3% while the manganese content could reach up to 0.4%. Machining and welding are relatively easy due to their high ductility. They are cheap and used more than the other types of carbon steel (Capudean, B. 2003). The pipelines steel have a typical range of various of API such as API 5L X60 (Mishra, 2014).

2.1.2.1 API 5LX60 Steel

In general, the pipelines are made of micro alloyed steel having properties commensurate with the standards of the American Petroleum Institute (API). These steels generally are intended for applications with dominant consideration of the efficiency to cost ratio with profitable weight reduction in wall thickness. These steels must have improved tensile strength, yield to tensile strength ratio, elongation, weld ability, susceptibility to hydrogen induced cracking particularly for sour service applications, low temperature impact toughness and ductile to brittle transition temperature (Mishra, 2014). The API steels are characterized on the basis of their leading micro alloying constituent and their effect during thermo-mechanical process. In the course of recent years, the trends towards expanded transportation effectiveness have to a great extent accomplished by expanding the width of pipelines. The properties of the pipe line steel needs to be equivalent with the measure of the standards of the American Petroleum Institute (API). The API steels are of different grades depending on the composition, properties and thickness. The properties in API steels are essentially obtained by addition of various micro alloying components (Heness & Cortie, 2012) for example Ti, B, P, N, S, Mn, V, Nb etc. and so forth the controlled TMP and cooling. The properties of the pipeline steels may be characterized as blend of strength, fracture toughness and weld ability, which is accomplished through thermo-mechanical, controlled processing (TMCP). The highest grade pipeline in commercial development today is X-100,consistent with historical trends as shown in Figure 2.1.

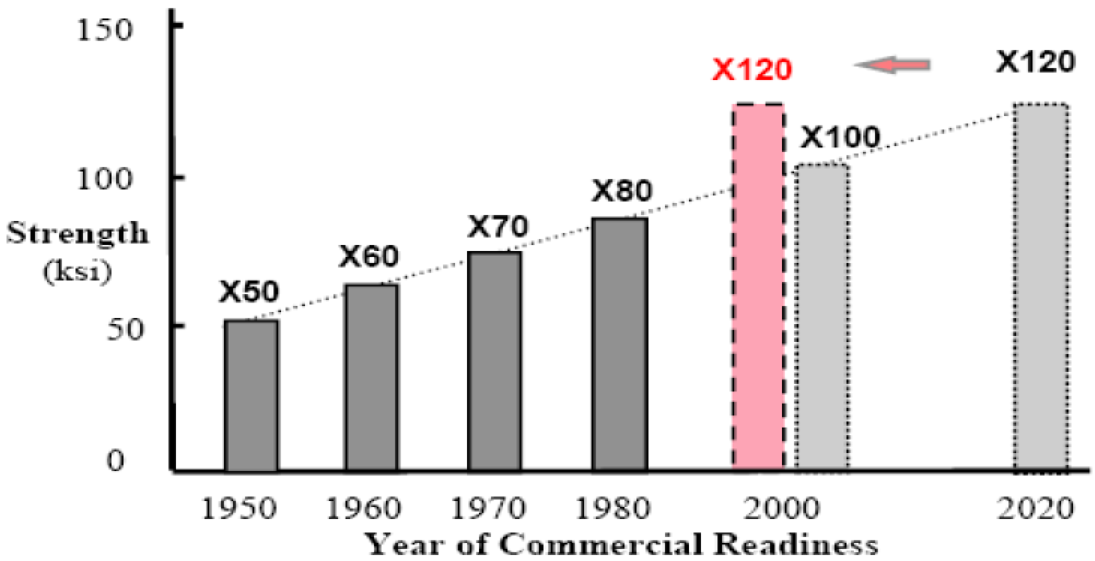


Figure 2.1: Commercialization of pipeline technology (Koo et al., 2004)

The API 5LX60 code means the steel has a minimum yield strength of 60ksi, which corresponding to approximately 414MPa. Table 2.2 shows the chemical composition and mechanical properties of API 5L X60 pipeline steel (Sunny-steel, 2011).

Table 2.2: Chemical composition and mechanical properties of API 5L X60 Steel

Chemical composition							
C	**SI**	**Mn**	**p**	**s**	**v**	**Nb**	**Ti**
≤0.28	≤0.45	≤1.60	≤0.03	≤0.01	≤0.15	≤0.05	≤0.04

Mechanical properties		
Tensile strength [MPa]	**Yield strength [MPa]**	**Elongation [%]**
≥435	≥320	≥28

2.2 Mechanical Testing

2.2.1 Impact Testing

In general, testing of materials can be done for the following purposes:

- To assess numerically the fundamental mechanical properties of ductility, malleability, toughness, etc.
- To determine data, i.e., force-deformation values to draw up sets of specifications upon which the engineer can base his design.
- To determine the surface or sub-surface defects in raw materials or processed parts.
- To check chemical composition.
- To determine suitability of a material for a particular application.

Testing on materials may involve destructive tests and/or non-destructive tests. In a destructive test, the components or specimen either breaks or remains no longer useful for future use. Examples of destructive tests are tensile test, impact test, bend test, torsion test, fatigue test, etc. A component or specimen does not break in non-destructive testing and even after being tested so, it can be used for the purpose for which it was made. Examples of non-destructive tests are radiography, ultrasonic inspection, etc. Impact is defined as the resistance of a material to rapidly applied loads. An impact test is a dynamic test in which a selected specimen which is usually notched is struck and broken by a single blow in a specially designed machine. The purpose of impact testing is to measure an object's ability to resist high-rate loading. It is usually thought of in terms of two objects striking each other at high relative speeds. A part or material's ability to resist impact often is one of the determining factors in the service life of a part, or in the suitability of a designated material for a particular application. Impact resistance can be one of the most difficult properties to quantify. The ability to quantify this property is a great advantage in product liability and safety. An impact test signifies toughness of material that is ability of a metal to deform plastically and to absorb energy in the process before fracture is termed toughness. The emphasis of this definition should be placed on the ability to absorb energy before fracture. Recall that ductility is a measure of how much something deforms plastically before fracture, but just because a material is ductile does not make it tough. The key to toughness is a good combination of strength and ductility. An impact test signifies toughness of material that is ability of a metal to deform plastically and to absorb energy in the process before fracture is termed toughness. The emphasis of this definition should be placed on the ability to absorb energy before fracture. It can be remembered that ductility is a measure of how much something deforms plastically before fracture, but just because a material is ductile does not make it tough. The key to toughness is a good combination of strength and ductility. A material with high strength and high ductility will have more toughness than a material with low strength and high ductility. There are several variables that have a profound influence on the toughness of a material. These variables are strain rate (rate of loading), temperature, notch effect. A metal may possess satisfactory toughness under static loads but may fail under dynamic loads or impact. Toughness decreases as the rate of loading increases. Temperature is the second variable to have a major influence on its toughness. As temperature is lowered, the ductility and toughness also decrease. The third variable is termed notch effect, has to do with the distribution of stress. A material might display good toughness when the applied stress is uniaxial; but when a multiaxial stress state is produced due to the presence of a notch, the material might not withstand the simultaneous elastic and plastic deformation in the various directions.

The essential features needed to perform proper impact test are:

- A suitable specimen (specimens of several different types are recognized),
- An anvil or support on which the test specimen is placed to receive the blow of the moving mass,
- A moving mass of known kinetic energy which must be great enough to break the test specimen placed in its path, and
- A device for measuring the energy absorbed by the broken specimen.

The main objective of the impact test is to predict the likelihood of brittle fracture of a given material under impact loading. The test involves measuring the energy consumed in breaking a notched specimen when hammered by a swinging pendulum. The presence of a notch simulates the pre-existing cracks found in large structures. Note that both impact loading and the presence of a notch increase the probability of brittle fracture. The energy absorbed can be calculated by measuring the change in the potential energy of the pendulum before and after breaking the specimen. ASTM has standardized the impact test with two testing approaches: the Charpy and the Izod (Gupta, 2015). The two tests differ mainly in how the specimen is supported during impact loading. In the Charpy test the specimen is supported as a simple-beam while in the Izod test the specimen is supported as a cantilever-beam (Gupta, 2015). Both tests use square bar specimens with machined notches taking the shape of the letter V hence giving other common names for these tests as Charpy V-notch (CVN) or Izod V-notch. Using an impact machine, the energy absorbed while breaking the specimen is measured (Ali et al., 2013). The energy quantities determined are qualitative comparisons on a selected specimen and cannot be converted to energy figures that would serve for engineering design calculations. The purpose of the impact test is to measure the toughness, or energy absorption capacity of the materials (Sawhney, 2009). In addition to providing information not available from any other simple mechanical test, these tests are quick and inexpensive. The data obtained from such impact tests is frequently employed for engineering purposes. It is usually thought of in terms of two objects striking each other at high relative speeds. It is usually employed to test the toughness of metals, but similar tests are used for polymers, ceramics and composites.

Pendulum impact machines consist of a base, a pendulum of either single-arm or "sectorial" design, and a striker rod (also called a hammer), whose geometry varies in accordance with the testing standard (see Figure 2.2). The mass and the drop height determine the potential energy of the hammer. Each pendulum unit has provisions to add extra weight. There is also a specimen support a vise for the Izod test and an anvil for the Charpy test. The principal measurement from the impact test is the energy absorbed in fracturing the specimen. After breaking the test bar, the pendulum rebounds to a height which decreases as the energy absorbed in fracture increases. The energy absorbed in fracture, usually expressed in joules, is rend directly from a calibrated dial on the impact tester.

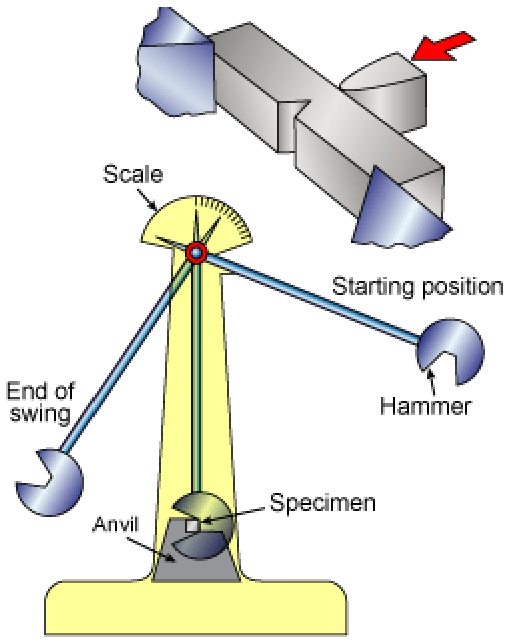


Figure 2.2: Pendulum Impact Machine (Laryee, 2018)

Izod and Charpy impact tests are similar in many respects (Singh, 2009). Both use test specimens that are either molded to size or cut from a larger "dog-bone" tensile-test sample. The test specimens have different dimensions. Specimen size (T×W×L) for Izod testing is 10 x10 x 75 mm, while Charpy uses 10 x 10 x 60 mm specimens. In both tests, sample thickness depends on the specifications for the material being tested (typically 1/8 in. for Izod tests). Specimens are notched and conditioned with temperature and humidity before testing. At least 3 specimens are tested and the results are averaged. The test notches for the impact specimens for the tests have different dimensions. The Izod test is a V-notch; the Charpy test has three different specimen types: Key-hole, U-notch, and V-notch. However, other specimen types may be specified as required for both tests. The specimens are held differently. The Izod specimen is held in a cantilevered manner; the Charpy test is held such that the specimen rests against two supports on either side of the test notch. The impact location is different. The Izod test impact is against the end of the exposed cantilever; the Charpy test is struck directly behind the test notch such that the specimen undergoes three point bending. Notches cut away a V-shaped section of the sample. The notch size and shape are specified by the test standard. The purpose of the notch is to mimic part-design features that concentrate stress and make crack initiation easier under impact loads. Notch toughness is the ability that a material can have to absorb energy in the presence of a flaw.

In the presence of a flaw, such as a notch or crack, a material will likely exhibit a lower level of toughness. When a flaw is present in a material, loading induces a triaxial tension stress state adjacent to the flaw. The material develops plastic strains as the yield stress is exceeded in the region near the crack tip. However, the amount of plastic deformation is restricted by the surrounding material, which remains elastic. When a material is prevented from deforming plastically, it fails in a brittle manner. The units of this property are reported in the literature as foot-pounds (ft-lb) in the English system and joules (J) in the metric system. ISO and ASTM standards express impact strengths in different units. ISO standards report impact strengths in kJ/m^2, where the impact energy is divided by the cross sectional area at the notch. ASTM standards call for values to be reported in J/m, where the impact energy is divided by the length of the notch. Units are ft-lb/in. for Izod and $joule/m^2$ for Charpy.

2.2.1.2 Impact Energy

Impact energy is a measure of the work done to fracture a test specimen. When the striker impacts the specimen, the specimen will absorb energy until it yields. At this point, the specimen will begin to undergo plastic deformation at the notch. The test specimen continues to absorb energy and work hardens at the plastic zone at the notch. When the specimen can absorb no more energy, fracture occurs. Notched impact data cannot be compared with unnotched. Brittle materials generally have lower impact strengths, while those registering higher impact strengths tend to be tougher.

Drop Weight Testing -this test is conducted to determine the zero ductility transition temperature (NDT) of materials. Dynamic Tear Testing has a wide range of Research and Development applications. Used to study the effects of metallurgical variables like heat treatment, composition, and processing methods on the dynamic tear fracture resistance of material. Manufacturing processes, such as welding, can be effectively evaluated for their effect on dynamic tear fracture resistance. Additional uses for this test include evaluating the appropriateness of selecting a material for an application where a baseline correlation between Dynamic Tear energy and actual performance has been developed.

2. 2.1.3 Impact Specimens

The testing of full sized parts or structures in impact is very difficult because of the magnitude of the force required to produce failure. Generally, notch type specimens are used for impact tests. The presence of a notch on the surface of the test area of the specimen creates a concentration of stress or localization of strain during test. The effect of the localized strain at the base of the notch causes the specimen to fail through the plane at relatively low values of energy. Since the effect of the notch localizes the strain at its base, any change in the shape of the notch at its base will influence the impact value obtained. Therefore, the accuracy in the manufacturing of test specimen is most important. A high degree of precision is required in shaping the notch and locating the bottom of the notch with respect to the opposite surface of the specimen. The accuracy surface of the maintained in the manufacture of any type impact test specimen is plus or minus 0.001 inch.

There are two general types of notches used in the Izod and Charpy impact tests (bending impact tests). These are classified as the keyhole notch and the V- notch. The keyhole notch is used only in the Charpy impact specimens and chief characteristic is the large radius at the root of the notch (0.039 inch radius). The V-notch has a small radius at the root of the notch (0.010 inch radius) and is used in both Charpy and Izod impact specimens. Another difference is the depth of the notch. In any notch-tough material, the V-notch specimens will give higher Charpy impact values than are obtained for the keyhole notched specimens because of the larger cross section of the material under test. However, when the material is not notch-tough, both types of specimens will give the same approximate Charpy impact values. According to ASTM A370 (Standard Test Method and Definitions for Mechanical Testing of Steel Products) Standard specimen for Charpy impact test is:

10mm×10mm×55mm. Sub size specimens are: 10mm×7.5mm×55mm, 10mm×6.7mm ×55mm, 10mm×5mm× 55mm, 10mm×3.3mm×55mm, 10mm×2.5mm×55mm.

2.2.1. 4 The Major Factors that Affect the Results of an Impact Test

The major parameters that influence the results of an impact test are

a) Velocity
b) Specimen

c) Temperature

a) Velocity

The velocity at impact does not appear to appreciably affect the results. However, experiments conducted with machines that develop velocities above certain critical values, impact resistance appears to decrease markedly. In general, the critical velocities are much less for annealed steels than for the same steels in the hardened condition.

b) Specimen

In some cases it is not possible to obtain a specimen of standard width from the stock that is available. Decreasing either the width or the depth of these specimens decreases the volume of metal subject to distortion, and thereby tends to decrease the energy absorption when breaking the specimen. The effect of the notch is to concentrate stresses at the root of the notch, embrittle the material in the vicinity of the notch and, at the same time, raise the elastic limit of the material in this area. When a crack forms at the root of the notch the stress is greatly intensified and the crack quickly progresses across the section. Without the notch, many compositions would simply bend without fracture, and their total capacity to absorb energy could not be detected. The sharper the notch (i.e. the smaller the included angle) the more pronounced are the effects noted above. The specimen sizes have been standardized so that results can be compared with reasonable confidence

c) Temperature

In contrast to the relatively small effect of temperature on the static strength and ductility of metals, at least within the atmospheric range, temperature has a very markedly effect on the impact resistance of the notched bars. Figure 2.3 shows the effect of temperature on the impact energy absorbed.

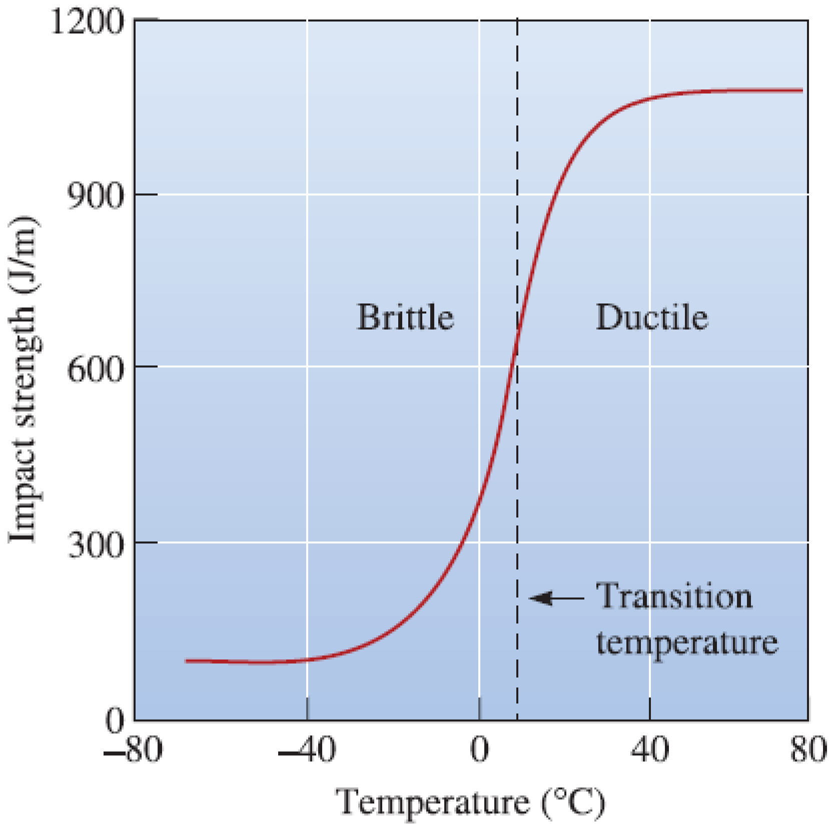


Figure 2.3: Effect of Temperature on the Impact Energy Absorbed (Askeland et al., 2006)

For a particular metal and type of test, below some critical temperature the failures are brittle, with low energy absorption. Above some critical temperature, the failures are ductile, with energy absorption that may be many times that in the brittle fracture range. Between these temperatures is what has been termed as transition-temperature range, where the character of the fracture may be mixed. With the standard notch, the critical range for many steels appears to occur between the freezing point and room temperature; in some metals it may be extended to temperatures well below the freezing point. Impact strength can be affected by temperature. This is especially true or carbon steels and other metals with a body-centered cubic (BCC) or hexagonal crystal (HCP) structure. Metals with a face-centered cubic (FCC) structure (such as austenitic stainless steel, copper, and aluminum) strengthen slightly at low temperatures, but there is not a significant lowering of impact strength as can be the case with carbon steels.

Furthermore, factors that affect the Charpy impact energy of a specimen (Gambhir & Jamwal, 2014) will include:

a) Yield strength and ductility
b) Notches
c) Temperature and strain rate
d) Fracture mechanism

a) Yield strength and Ductility

For a given material the impact energy will be seen to decrease if the yield strength is increased, i.e. if the material undergoes some process that makes it more brittle and less able to undergo plastic deformation. Such processes may include cold working or precipitation hardening.

b) Notches

The notch serves as a stress concentration zone and some materials are more sensitive towards notches than others. The notch depth and tip radius are therefore very important.

c) Temperature and Strain rate

Most of the impact energy is absorbed by means of plastic deformation during the yielding of the specimen. Therefore, factors that affect the yield behavior and hence ductility of the material such as temperature and strain rate will affect the impact energy. This type of behavior is more prominent in materials with a body centered cubic structure, where lowering the temperature reduces ductility more markedly than face centered cubic materials.

d) Fracture mechanism

Metals tend to fail by one of two mechanisms, micro void coalescence or cleavage. Cleavage can occur in body centered cubic materials, where cleavage takes place along the {001} crystal plane. Micro void coalescence is the more common fracture mechanism where voids form as strain increases, and these voids eventually join together and failure occurs. Of the two fracture mechanisms cleavage involved far less plastic deformation ad hence absorbs far less fracture energy

2. 2.1.5 Theoretical Explanation of Pendulum Test

In a typical Pendulum machine (Figure 2.4), the mass of the hammer (striking edge) mass (m) is raised to a height (a). Before the mass (m) is released, the potential energy will be (Singh, 2009):

$$E_p = mga \qquad (2.1)$$

After being released, the potential energy will decrease and the kinetic energy will increase. At the time of impact, the kinetic energy of the pendulum (E_k)

$$E_k = \frac{1}{2}mv^2 \qquad (2.2)$$

And the potential energy:

$$E_p = mga \qquad (2.3)$$

Will be equal, $E_k = E$

$$mga = \frac{1}{2}mv^2 \qquad (2.4)$$

$$v^2 = 2\,ga \qquad (2.5)$$

And the impact velocity will be:

$$v = \sqrt{2\,ga} \qquad (2.6)$$

hammer continues its upward motion but the energy absorbed in breaking the test piece reduces its momentum. A graduated scale enables a reading to be taken of the energy used to fracture the test piece. To obtain a representative result the average of three tests is used and to ensure that the results conform to those of the steel specification the test specimens should meet the standard dimensions. This test can also used to determine the notch sensitivity.

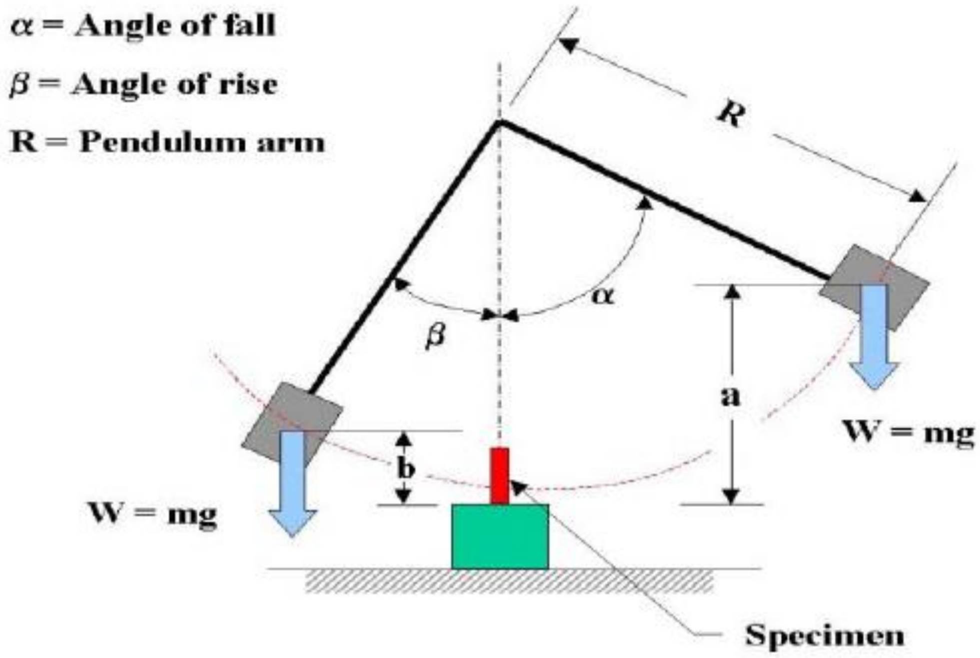


Figure 2.4: Typical Pendulum Machine (Darvell, 2009)

$$a = R(1 - cos\alpha) \quad (2.7)$$

$$b = R(1 - cos\beta) \quad (2.8)$$

Initial energy (E_i)

$$E_i = mgR(1 - cos\alpha) = WR(1 - cos\alpha) \quad (2.9)$$

Energy after the rupture (E_r)

$$E_r = mgR(1 - cos\beta) = WR(1 - cos\beta) \quad (2.10)$$

Energy absorbed by the specimen (E_{abs})= E_{abs} =W R (cos β - cos α)

$$E_i = WR(cos\alpha - cos\beta) \quad (2.11)$$

2. 2.1. 6 Izod Impact Test

The Izod Impact Test (Figure 5) was invented by Edwin Gilbert Izod (1876-1946). A test specimen, usually of square crossed section is notched and held between a pair of jaws, to be broken by a swinging or falling weight. When the pendulum of the Izod testing machine is released it swings with a downward movement and when it reaches the vertical the hammer makes contact with the specimen which is broken by the force of the blow. The

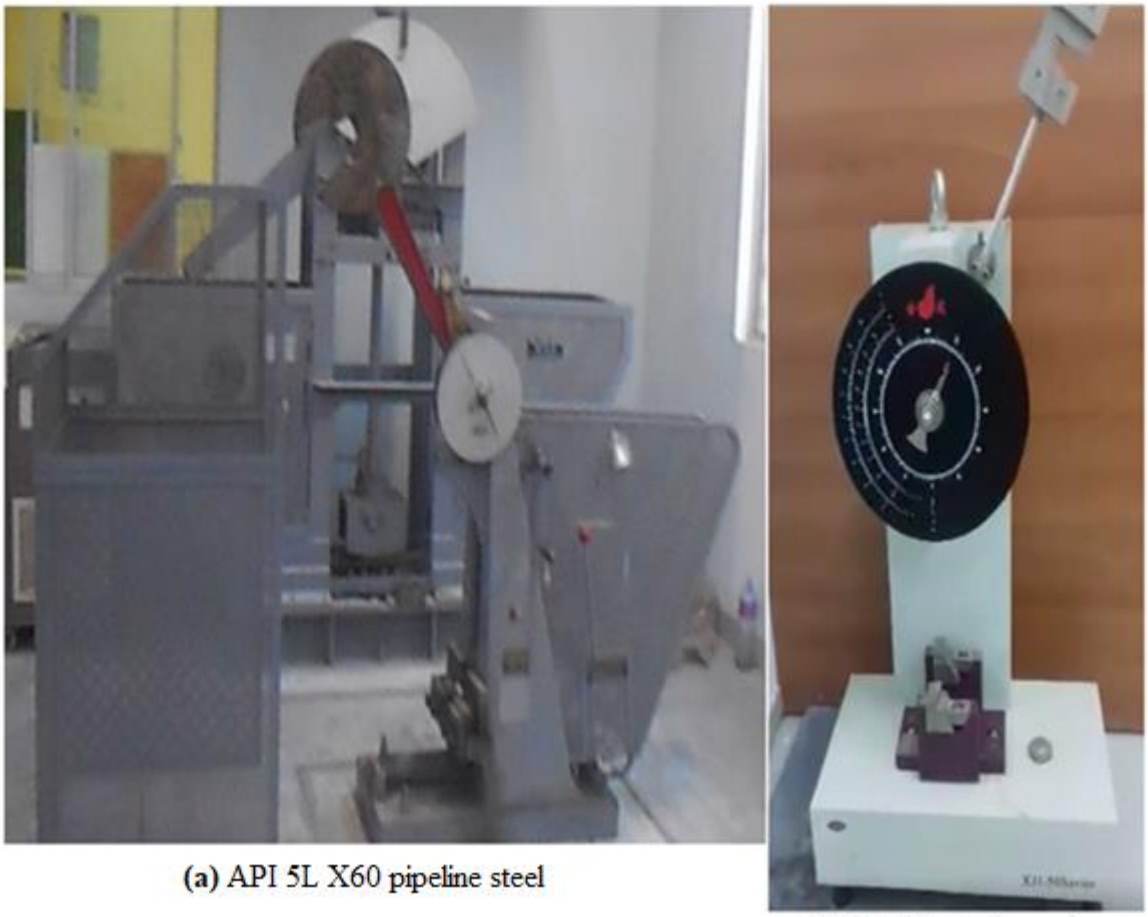
Figure 2.5: Impact Testing Machine (Izod) available in Libyan Iron and Steel Company, Misurata, Libya

This impact testing machine is capable of performing both Izod and Charpy impact test. This has separate hammers for both tests, a vice for Izod test and an anvil for the Charpy test to hold the specimen according to standard specimen size, height of hammer, separate scale and other accessories to perform both impact test. It is used for the purpose of performing Izod test in solid mechanics lab at company. Where separate impact testing machine is used to perform Charpy impact test. Izod testing can be done up to 0 to 164

Joules or N-m. The testing equipment is the impact testing shown in figure No. 2.4 where the fracture energy in Joules can be read directly from the dial on the tester for both Izod and Charpy impact test. The test specimen is machined to a square or round section, with either one, two or three notches. The specimen is clamped vertically on the anvil with the notch facing the hammer. The Izod test is has become the standard testing procedure for comparing the impact resistances of plastics. While being the standard for plastics it is also used on other materials. The Izod test is most commonly used to evaluate the relative toughness or impact toughness of materials and as such is often used in quality control applications where it is a fast and economical test. It is used more as a comparative test rather than a definitive test. This is also in part due to the fact that the values do not relate accurately to the impact strength of moulded parts or actual components under actual operational conditions. When releasing the pendulum and make sure to clear the way and stand back away from the swinging pendulum.

2. 2.1.7 Charpy Impact Test

The Charpy Impact Test was developed in 1905 by the French scientist Georges Charpy (1865-1945) (Tóth et al., 2002; Westmoreland Mechanical Testing & Research, 2002). The Charpy test measures the energy absorbed by a standard notched specimen while breaking under an impact load (Wright & Askeland, 2016). The Charpy impact test continues to be used as an economical quality control method to determine the notch sensitivity and impact toughness of engineering materials (Wright & Askeland, 2016). The Charpy Test is commonly used on metals, but is also applied to composites, ceramics and polymers (Figure 2.6). With the Charpy test one most commonly evaluates the relative toughness of a material, as such; it is used as a quick and economical quality control device. It was pivotal in understanding the fracture problems of ships during the Second World War, Today it is used in many industries for testing building and construction materials used in the construction of pressure vessels, bridges and to see how storms will affect materials used in building.



(a) API 5L X60 pipeline steel

(b) HDPE pipeline polymer

Figure 2.6: Charpy Impact Testing Machine with specimens

Charpy pendulum impact testing machine has eighteen numbers of teeth. The pendulum can be raised up to fifteen teeth. It measures impact energy absorbed in Kg-m. The potential energy of hammer is increased 2.5 Kg-m by increase in each teeth. A Charpy pendulum impact test is a variation of Izod. In a Charpy test, a sample is laid horizontally on two supports against an anvil. The sample is notched in the center and the notch side is positioned away from the pendulum. When the pendulum swings through the gap in the anvil, it impacts the center of the sample with a hammer. The energy to break is measured and reported in the same way as with an Izod test. The principal difference between two tests is the manner in which the specimen is supported. This position places the notch at the location of the maximum tension.

The standard test specimen is 10 x 10 x 55 mm, with a v-notch 2 mm deep on one side at the center. The specimen is placed exactly midway between two anvils such that the pendulum strikes opposite to the notch. The pendulum is lifted to the initial release position and then released. The pendulum must be allowed to swing freely after striking the specimen. When releasing the pendulum and make sure to clear the way and stand back away from the swinging pendulum. Do not try to stop the pendulum once it has been released. It can cause serious injury. The standard Charpy Test specimen consist of a bar of metal, or other material, 55x10x10mm having a notch machined across one of the larger dimensions. Figure 2.7 and 2.8 show the dimensions of the Charpy test specimen and the positions of the striking edge of the pendulum and the specimen in the anvil.

V-notch: 2mm deep, with 45° angle and 0.25mm radius along the base.

U-notch and keyhole notch: 5mm deep notch with 1mm radius at base of notch.

Figure 2.7: Charpy Impact Test specimens (WMTR, 2018)

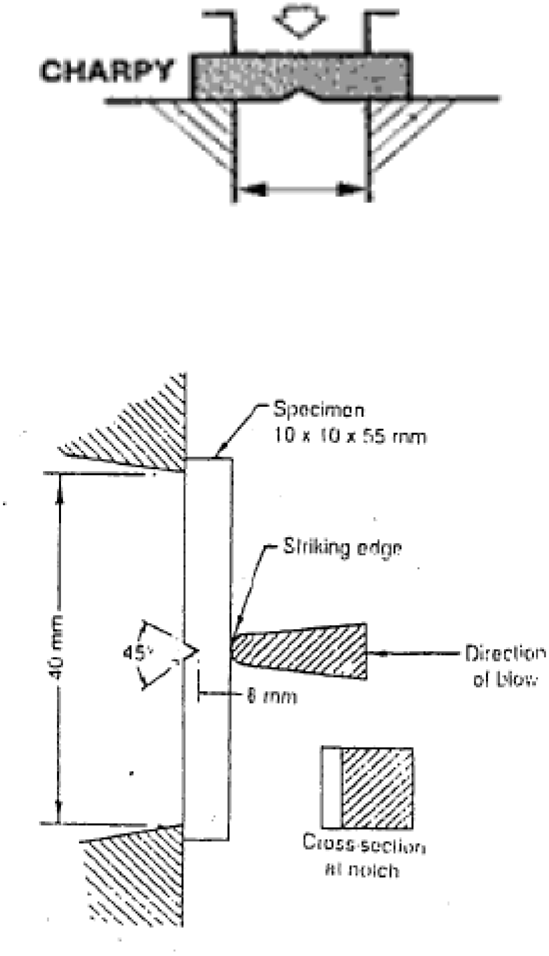


Figure 2.8: Position of the Charpy test specimen on the impact test (Darvell, 2009)

The Charpy tests are conducted on instrumented machines capable of measuring less than 1ft.lb. to 300ft. lbs. at temperatures ranging from - 320°F(0ºC) to over 2000°F. Specimen types include notch configurations such as V-Notch, U-Notch, Key-Hole Notch, as well as Un-notched and ISO (DIN) V-Notch, with capabilities of testing sub size specimens down to 1/4 size. A test specimen is machined to a 10mm x 10mm (full size) cross-section, with either a "V" or "U" notch. Sub-size specimens are used where the material thickness is restricted. Specimens can be tested down to cryogenic temperatures.

2.2.2 Tension and Hardness Test

The testing of thermoplastics to obtain data for the simulation of the in-service mechanical performance of thermoplastic components and the correlation with test results is not well understood by the majority of the thermoplastics industry. This has resulted in the majority of mechanical testing being done to compare materials and not to supply data for design purposes. Thus despite there being a past conference targeted to solve this problem the role of materials testing within the design and development process is still being debated. Documents such as those supplied by BASF and Hoechst Celanese (Hoechst, 1991; Müller, 1981) provide an insight into material testing methods and basic design methods for thermoplastic parts (Bannantine et al., 1990). These, however do not provide quantitative information on how to predict the impact performance of thermoplastic components. The required material properties for an impact simulation of a thermoplastic component are those which can be used to define a three dimensional material model describing the stress-strain curve to failure. However there is no one single test configuration which is suitable for testing materials in all three orthogonal axes and in both tension and compression. Furthermore it is normal to characterize the stress-strain curve of a material by a number of nominal parameters, for example initial low strain elastic modulus (E), stress at the onset of neck formation (σ_n) and strain to onset of neck formation (ε_n). The ideal test method would be quick, accurate, insensitive to sample preparation and low cost. However all test methods have limitations and prior to using material stiffness and strength measurements it is necessary to understand how they were measured. In general there are three basic methods for measuring polymeric material stiffness and strength: quasi static, creep/relaxation and dynamic which are reviewed briefly. For all tests it should be noted that although it is desired that test samples are normally subjected to one dimensional quasi static loads both test samples and test equipment are three dimensional objects and have distributed mass, stiffness and damping. Thus there are always the potential undesired complications due to deviations from one dimensional to three dimensional specimen loading and quasi static to dynamic loading of both specimen and test equipment.

Illustrated in Figure 2.9 this is the most widely used method to mechanically test materials. Capable of recording the whole of the engineering stress-strain curve, the standard output parameters that are quoted from this test are the engineering measures of E, σ_n and ε_n, (BS

2782-3, 1976). A wide range of test strain rates can be achieved by the use of electric motors and screw driver or hydraulic actuator drive mechanisms. Running servo hydraulic testing machines at increasing strain rates has several notable drawbacks. At high strain rates, e. g. above $20s^{-1}$, there is a likelihood of producing erroneous data due to "inertial effects and load cell ringing" (Nicholas, 1981). Thus instead of measuring a quasi static event the experimenter measures the inertia response of the test equipment. In this test the sample shape has been designed to minimize the effects of holding the sample such that the desired applied stress field in the gauge length is approximately constant. As the cross-head displacement is not necessarily the same as the sample strain it is necessary to instrument the specimen and use this in a closed loop control circuit to achieve a constant strain rate. Instrumentation on the gauge length of a thermoplastic test sample is difficult as contacting extensometers can cause premature failure of the sample at their attachment points, non-contacting extensometers may have insufficient resolution to accurately determine low strain modulus and strain gauges may adhere poorly due to the low surface activity of the thermoplastics. In the British standard, BS, (BS 2782-3, 1976), the material properties that are normally evaluated are initial low strain secant modulus, and engineering stress and strain at the point of maximum load during the test. At the point of maximum load the material sample begins to neck and the stress-strain field in the gauge length becomes non-uniform. To measure the stress and strain beyond this point is extremely difficult and not normally done. Attempting to reconcile the difference between one dimensional and three dimensional stress-strain fields, non-contacting video cameras have been used to determine the shape of the neck zone and hence deduce the stress and strain in the neck (BS 2782-3, 1978) by applying three dimensional stress concentration factors and calculating an equivalent one dimensional stress from the three dimensional neck zone. This approach is potentially flawed as a prior knowledge of the stress concentrations and relationship between one and three dimensional behaviour is required.

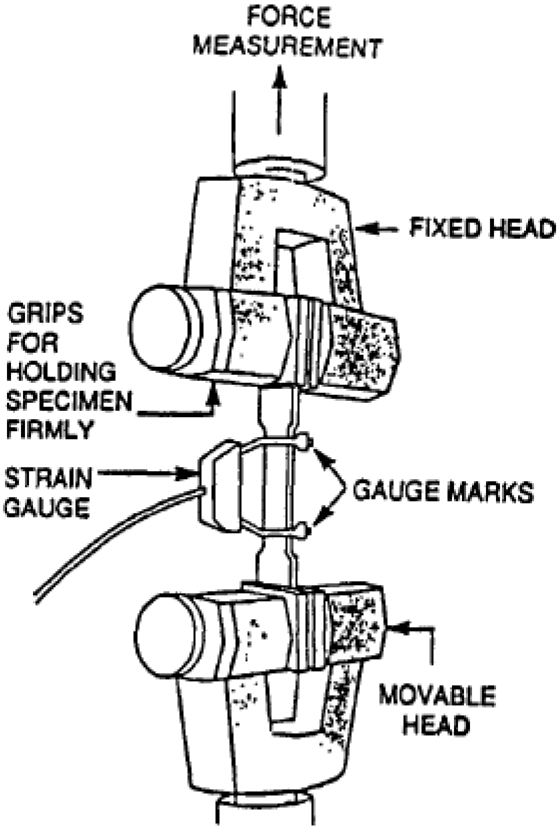


Figure 2.9: Tensile test geometry (Coulton, 1996)

2.2.2.1 Definition of stress and strain

a) **Engineering Stress and Strain**

There are several commonly used definitions of stress and strain (Bannantine et al., 1990). During the early 1980's it was the author's experience that the aerospace industry would only undertake linear analyses to predict the static and dynamic performance of structures (Coulton, 1996). If it was predicted that either the applied load exceeded the proportional limit of the material or the deformation of the structure was gross and thus likely to be non-linear then the design was deemed to have "failed" by exceeding the design brief. This design brief was set as non-linear analyses could not be achieved within the available cost and design cycle time scales. Thus, when it was necessary to characterize new material only the linear elastic material properties were measured with the onset of non-linearity being defined as failure (non acceptable material behaviour). The most common mechanical test undertaken for material samples is the tensile test. As this test has been developed for metals the common measurements from this test reflect the small strain nature of the properties that the test definers desired to measure. The standard way of converting load and deformation, in the tensile test, to stress and strain is to divide the load by the original cross sectional area of the specimen to obtain stress and divide the extension of the specimen by the original gauge length over which it was measured to obtain strain. These are known as the engineering or Cauchy definitions of stress and strain.

$$\sigma_{eng} = \frac{F}{A_o} \quad ; \; \varepsilon_{eng} = \frac{l - l_0}{l_0} = \frac{\Delta l}{l_0} \qquad (2.12)$$

Figure 2.10 shows the typical engineering's stress-strain curves measured, using a screw driven tensile test machine, for ductile and high strength concrete reinforcing bars.

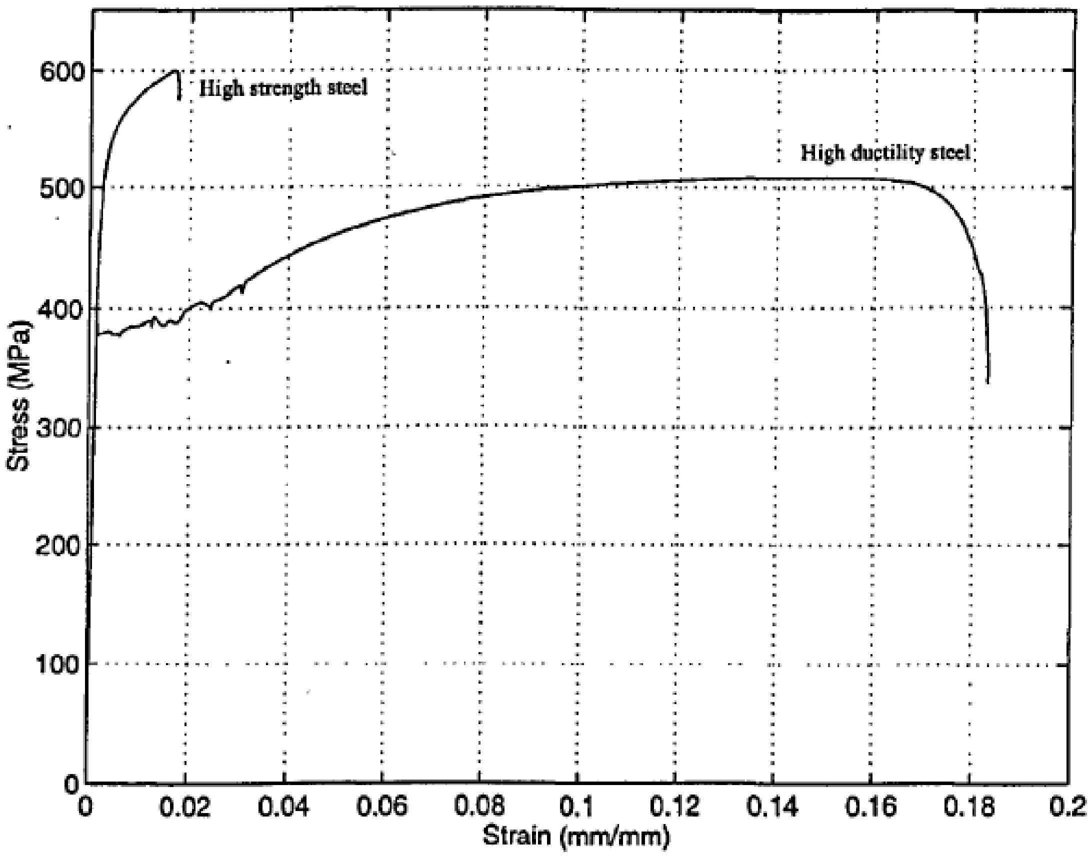


Figure 2.10: Typical engineering stress-strain curves of two different steels (Coulton, 1996)

Below the onset of yield the material stiffness is normally described by a modulus of elasticity defined as follows: "The modulus of elasticity, or Young's modulus, is the slope of the stress-strain curve in the elastic region. This relationship is Hooke's law" (Askeland, 1990)

$$E = \frac{\Delta\sigma_{eng}}{\Delta\varepsilon_{eng}} \tag{2.13}$$

Likewise the lateral strains induced in the tensile test are characterized by Poisson's ratio defined as the ratio of "longitudinal elastic deformation produced by a simple tensile or compressive stress to the lateral deformation that must simultaneously occur" (Askeland, 1990)

$$\nu = \frac{-\varepsilon_{lateral}}{\varepsilon_{longnitudinal}} \tag{2.14}$$

It should be noted that these are engineering measures and only applicable to small strain theory. If it is wished to go beyond small strain theory, which is generally the case when using thermoplastics, then the definition of stress and strain must be redefined to be mathematically correct. These are known as the true or Hencky definitions of stress and strain (Bannantine et al., 1990).

$$\sigma = \frac{F}{A} \quad ; \quad \varepsilon = \int_{l_0}^{l} \frac{dl}{l} = ln\left(\frac{l}{l_0}\right) \tag{2.15}$$

Similarly redefining of modulus of elasticity allows it to be non-linear and non-elastic.

$$E = \frac{d\sigma}{d\varepsilon} \tag{2.16}$$

As the applied strain approaches zero, the true definitions for stress, strain and modulus approach their engineering definitions.

b) True Stress and Strain

If the results of tensile testing are to be used to predict how a metal will behave under other forms of loading, it is desirable to plot the data in terms of true stress and true strain.

True stress (σ) is defined as

$$\sigma = \frac{F}{A} \tag{2.17}$$

where A is the cross-sectional area at the time that applied force is F. Up to the point at which necking starts, true strain (ε) is defined as

$$\varepsilon = ln\left(\frac{L}{L_0}\right) \quad (2.18)$$

where L_0 is the initial gage length and L is the new gage length.

This definition arises from taking an increment of true strain, $d\boldsymbol{\varepsilon}$, as the incremental change in length, dL, divided by the length , L, t the time, $d\boldsymbol{\varepsilon}=dL/L$, and integrating. As long as the deformation is uniform along the gage section, the true stress and strain can be calculating from the engineering quantities.

True and engineering stress and strain are related according to Equations 2.18 and 2.19 are valid only to the onset of necking; beyond this point true stress and strain should be computed from actual load, cross-sectional area, and gauge length measurements.

$$\sigma_T = \sigma(1+\varepsilon) \quad (2.19)$$

$$\varepsilon_T = ln(1+\varepsilon) \quad (2.20)$$

where, σ_T and σ are true and engineering stress, respectively, ε_T and ε are true and engineering strain, respectively,

Figure 2.11 shows a comparison of typical tensile engineering stress–strain and true stress–strain behaviors. It is observed that necking begins at point M on the engineering curve, which corresponds to M on the true curve. The "corrected" true stress–strain curve takes into account the complex stress state within the neck region.

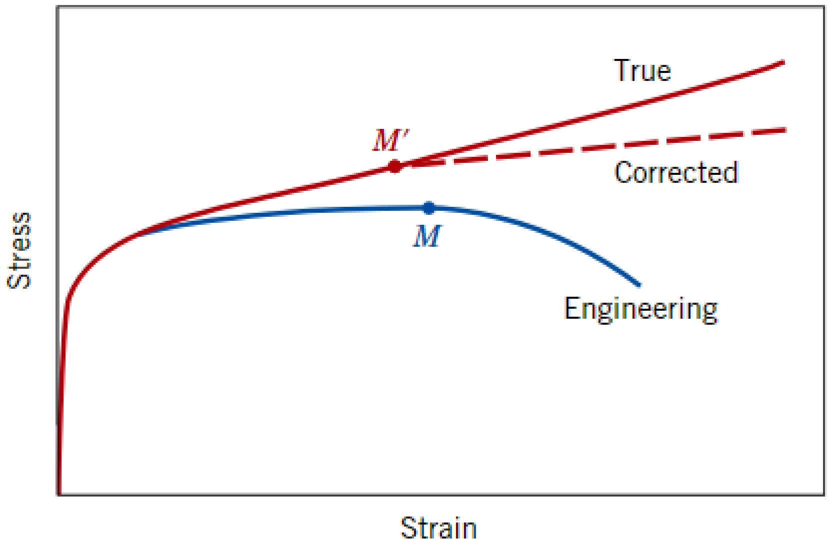


Figure 2.11: Engineering stress–strain and true stress–strain behaviors (Wright & Askeland, 2016)

8

2.2.3 Hardness Testing

Strength of a metal can be tested indirectly using a hardness test. In hardness test, a hard material called indenter is forced into material surface with some fixed load. Indenter makes an indent on the metal surface. Indent is defined by some number which expresses the hardness of the metal Verhoeven, 2007). Resistance of steel to indentation can be described as hardness of steel. Hardness measurement can be obtained using different methods (Javaherdashti & Tan, 2013):

- The Brinell Test that uses 10 mm- diameter ball indenter under a load of 29.420 N.
- The Vickers Test where the shape of the indenter is a diamond pyramid. Load can be changed.
- The Rockwell Test where the load is fixed -1471 N. Indenter is diamond cone.

There are some interrelationships between hardness and material. It is well known that hardness of metal alloys is higher than hardness of their individual components (Herrman, 2011).

2.2.4 Correlation between Hardness and Tensile Strength

Both tensile strength and hardness are indicators of a metal's resistance to plastic deformation. Consequently, they are roughly proportional, as shown in Figure 2.12, for tensile strength as a function of the HB of cast iron, steel, and brass. The same proportionality relationship does not hold for all metals, as Figure 2.12 indicates. As a rule of thumb for most steels, the HB and the tensile strength are related according to

$$TS = 3.45 \times HB \qquad \text{[MPa]} \qquad (2.21)$$

$$TS = 500 \times HB \qquad \text{[psi]} \qquad (2.22)$$

where, HB is Brinell hardness number and TS is tensile strength in MPa/psi.

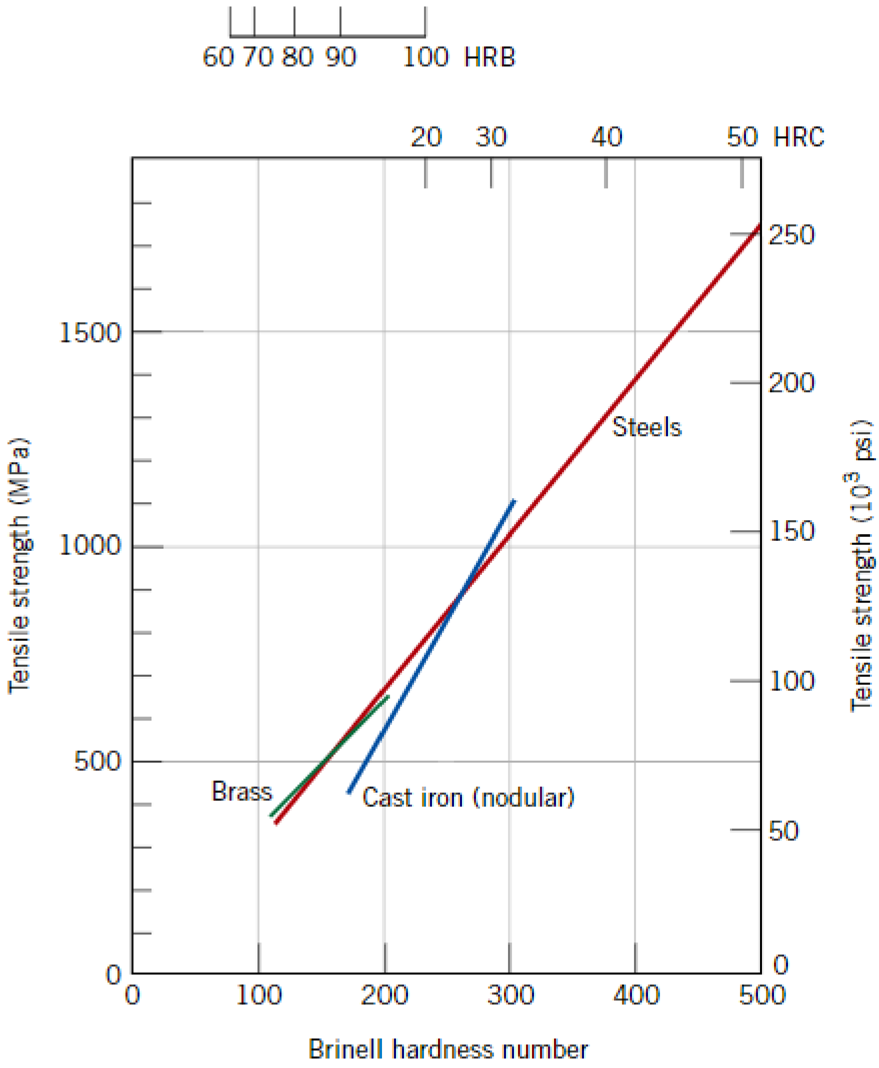


Figure 2.12: Relationships between hardness and tensile strength for different materials (Wright & Askeland, 2016)

2.3 Water-Jet Cutting Technique

The waterjet cutting technology is one of the most modern non-traditional cutting methods (Florén, 2011).. The principle of waterjet cutting is simple: compressed water is passed through a very small nozzle. The pressure inside the nozzle is transformed into kinetic energy and comes out as a thin water jet with a velocity of 900 m/s. Technique of cutting with water stream is called pure waterjet cutting and is able to cut through softer materials like food, rubber, plastic, wood. In order to get a higher cutting force abrasive waterjet cutting (AWJ) technology was developed where particles of a very hard abrasive medium (usually Garnet grains) are added to the waterjet. Figure 2.13 shows how an AWJ cutting head is build (Florén, 2011).

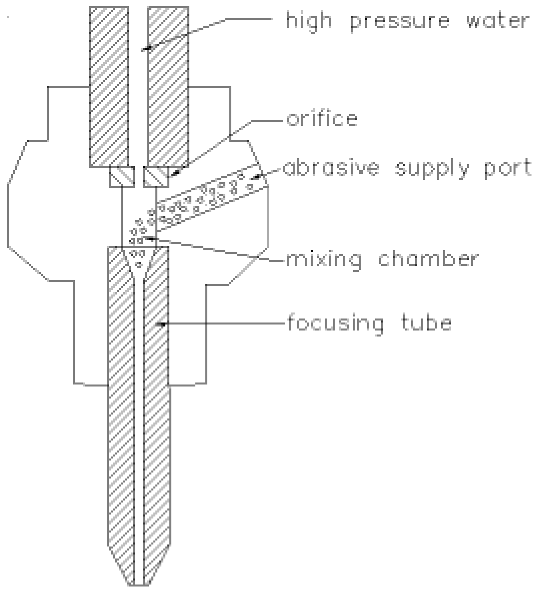


Figure 2.13: AWJ cutting head (Florén, 2011).

The high pressure waterjet released through an orifice is broken into small drops in the mixing chamber where these drops transfer energy to the abrasive particles. The abrasive waterjet becomes a steam of particles consisting of around 4% water, 1% abrasive grains and the rest is air. The erosive power of such mixture is able to cut practically any material (e.g. steel, stone, titanium, composite materials) even at great thicknesses (up to 300 mm thick steel and titanium are being cut) (Öjmertz, 2006).

The abrasive waterjet is a dynamic tool. The resulting AWJ kerf wall has a smooth surface at the upper part and changes gradually towards the lower part where striations and waviness appear. During cutting the jet moves dynamically influenced by two types of erosion processes interacting in material removal as well as oscillation caused by instability of the jet as it moves through the material. The striation appears because the jet loses its energy at increasing depth and becomes more unsteady. The instability of the jet may originate from pressure fluctuations or variations in particle distribution in the jet. It may also be result of inhomogeneous material which is the basis for an uneven resistance to erosion. Mechanical vibration transferred on the jet by the machine control system may also influence cutting stability (Florén, 2011).

2.4 Cryogenic Treatment

Cryogenic heat treatment is used commonly in allow and tool steel to improve wear resistance. When metals are subjected to cryogenic temperature (such as liquid nitrogen exposure at 190°C)

1) Their atoms lose kinetic energy, allowing the matrix to contract and ‘’work out’’ residual stresses and lattice imperfections from hot to cold working methods. This process gives parts tools the ability to last longer, resist cracking and be machined more easily.
2) Exposure to cryogenic heat treatment directly following a hardening procedure encourages the completion of martensitic phase transformation in steels. After a typical hardening process, a small residual percentage of soft austenitic remains, which may be undesirable for final properties.

The harder marten site phase fraction can be boosted nearly to 100 percent by heat treatment with liquid nitrogen for sufficient time (Davis, 1998).

Mechanical properties of materials depend on temperature (Figure 2.14). Yield strength, tensile strength, and modulus of elasticity decrease at higher temperatures, whereas ductility commonly increases. A materials fabricator may wish to deform a material at a high temperature (known as hot working) to take advantage of the higher ductility and lower required stress.

A high temperature is defined relative to the melting temperature. Thus, 500°C is a high temperature for aluminum alloys; however, it is a relatively low temperature for the processing of steels. In metals, the yield strength decreases rapidly at higher temperatures due to a decreased dislocation density and an increase in grain size via grain growth or a related process known as recrystallization. Similarly, any strengthening that may have occurred due to the formation of ultrafine precipitates may also decrease as the precipitates begin to either grow in size or dissolve into the matrix. When temperatures are reduced, many, but not all, metals and alloys become brittle. Increased temperatures also play an important role in forming polymeric materials

and inorganic glasses. In many polymer-processing operations, such as extrusion or the stretch-blow process, the increased ductility of polymers at higher temperatures is advantageous. Again, a word of caution concerning the use of the term “high temperature.” For polymers, the term “high temperature” generally means a temperature higher than the glass-transition temperature (T_g). The glass-transition temperature is not a fixed temperature, but depends on the rate of cooling as well as the polymer molecular weight distribution. Many plastics are ductile at room temperature because their glass-transition temperatures are below room temperature. To summarize, many polymeric materials will become harder and more brittle as they are exposed to temperatures that are below their glass-transition temperatures. The reasons for loss of ductility at lower temperatures in polymers and metallic materials are different.

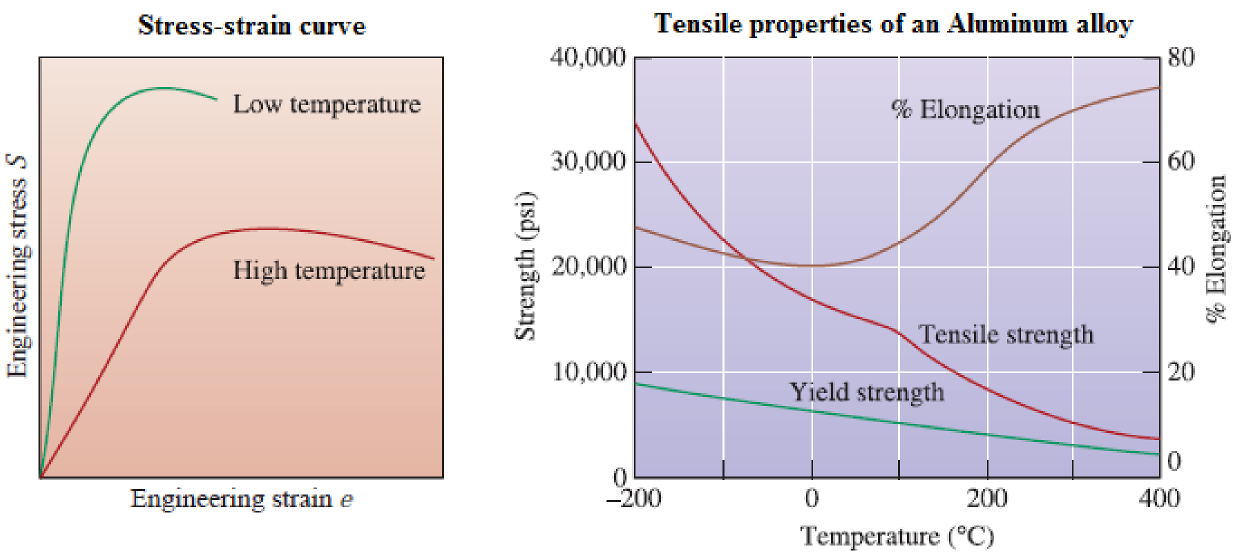


Figure 2.14: The effect of temperature on the stress–strain curve and on the tensile properties of an aluminum alloy (Askeland et al., 2006)

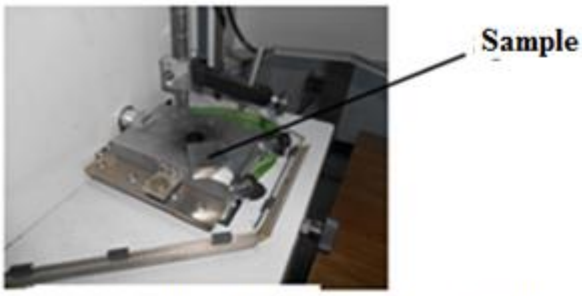
CHAPTER 3
EXPERIMENTAL WORK

3.1. Materials Used

Materials selected in this study were API 5L X60 steel and HDPE. API 5L X60 and HDPE are among the most common pipe material in Libya, Table 2.2, 3.1 and 3.2. All pipe materials used in this experiment were donated and subject to availability from the pipe manufacturers. The equipment used for machining all the samples was a high pressure waterjet machine; this type of machine is widely used in industries for cleaning, surface preparation, and cutting of soft materials (Figure 3.1). During the mechanical tests, API 5L X60 and HDPE pipe samples were first tested at temperature 25℃ (Room Temperature, RT), then they were cooled in liquid nitrogen temperature, NT, (-196℃) before tested. In this study, spectrometer analyzer (Figure 3.2) was used to obtain the material compositions of steel at both temperatures, RT (without liquid nitrogen treatment) and NT (with liquid nitrogen treatment). The nominal compositions of API 5L X60 are listed in Table 3.1, which shows the major elements of the API 5LX60 pipeline (full chemical analysis of API 5LX60 pipeline are presented in Appendix 1. It is concluded that testing temperature may have an effect the chemical composition of API 5L X60. Moreover, the material compositions of HDPE material are tabulated in Table 3.2. The liquid nitrogen was obtained from Libyan Iron and Steel Company at Misrata. It was possible to cool the samples down to -196℃ in the liquid Nitrogen.

Figure 3.1: Waterjet cutting machine

(a) Spectrometer analyzer



(b) Spectrometer analysis of a sample without liquid nitrogen treatment

(c) With liquid nitrogen treatment

Figure 3.2: Spectrometer analysis

Table 3.1. Major elements in the API 5L X60 pipeline steel found in the analysis

No. of test	Element			
	Fe	**C**	**Mn**	**Si**
	RT			
1	98.64216	0.15357	0.89711	0.18281
2	98.66088	0.15285	0.88419	0.17987
3	98.66805	0.1503	0.88184	0.17822
Average	98.65703	0.15224	0.887713	0.1803
SD	0.013366	0.001816	0.008219	0.002329
SD%	0.01	1.19	0.93	1.29
	NT			
1	98.7439	0.14326	0.84746	0.16841
2	98.727	0.13341	0.85412	0.16753
3	98.727	0.13266	0.85223	0.16005
Average	98.7228	0.136443	0.85127	0.16533
SD	0.007017	0.005912	0.003433	0.000968
SD%	0.01	4033	0.4	0.58

Table 3.2: Material properties of HDPE

RT		NT	
Density	≥930 kg/m^3	Density	≥930 kg/m^3
Melt Flow index	0.3 g/10 min	Melt Flow index	0.3 g/10 min
% black carbon	2 -2.5%	% black carbon	2 -2.5%
Young’s Modulus	0.55 -1 GPa	Young’s Modulus	0.55 -1 GPa
Yield Stress	20 – 30 MPa	Yield Stress	20 – 30 MPa
Stain at failure	≥ 350%	Stain at failure	≥ 350%
Shore hardness	39-40 N	Shore hardness	35-36 N
Toughness	2 – 5MPa.m$^{0.5}$	Toughness	2 – 5MPa.m$^{0.5}$

All standardized specimen samples were machined according to the standard given in Table 3.3. These standards specifies the necessary measurements, ratios and tolerances the test specimens must obtain in order to yield reliable results. Care was taken during machining to obtain as close to optimal measurements as possible.

Table 3.3: Standard used in this work

Tests	Pipeline material	Standards
Tensile stress testing	Low carbon steel	ASTM E 8M – 04
Tensile stress testing	HDPE	ISO 6259
Impact test	API 5L X60and HDPE	ASTM E 23 – 00
	HDPE	BS EN ISO 179:1997

3.2 Charpy Impact Testing

This test was used to investigate the behavior of the specimen under impact conditions.

3.2.1 Specimen Preparation

Test specimens for Charpy impact test are machined with small API 5L X60 and HDPE pieces according to ASTM E23 – 00 and BS EN ISO 179: 1997, respectively. According to the standard ASTM E23 – 00 (Appendix 3), the standard Charpy-V notch specimen of API 5L X60 should have dimensions as given in Figure 3.3, with a length of 55 mm and height and with both of 10 mm with a notch depth of 2 mm, an angle of 45° and a radius of curvature of 0.25 mm. While, the dimensions of the specimen of HDPE according to BS EN ISO 179: 1997standard for Charpy edgewise impact with single-notched and Charpy flat wise impact is shown in Figure 3.4.

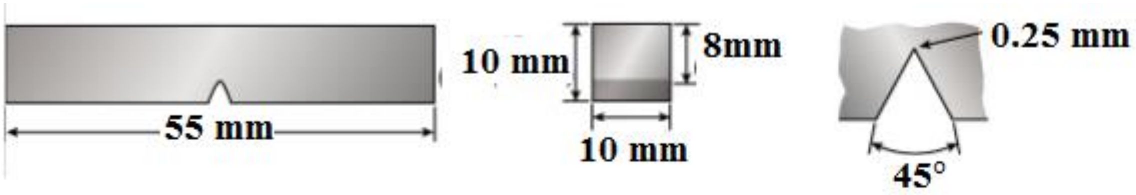
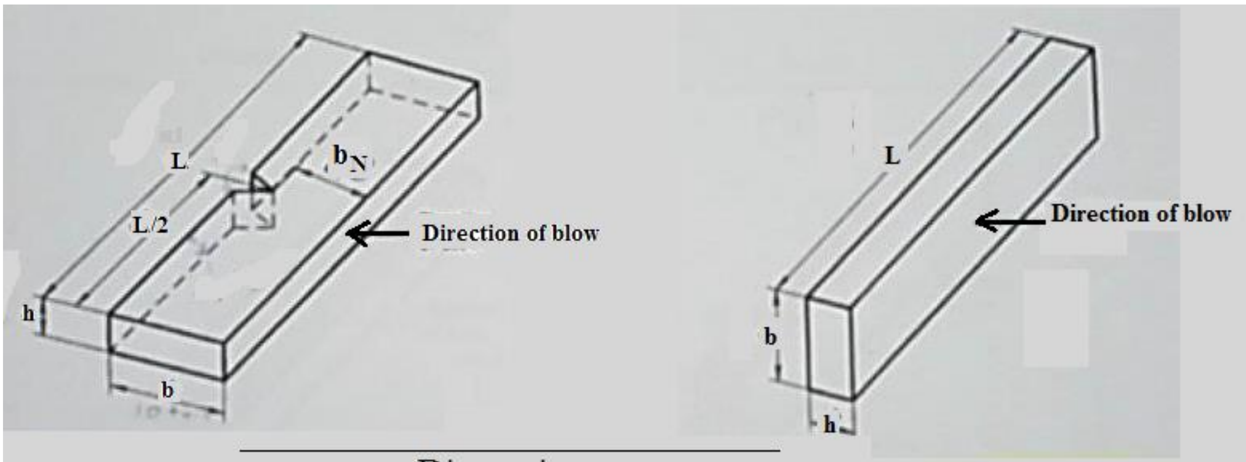


Figure 3.3: The standard Charpy test specimen of API 5L X60 steel

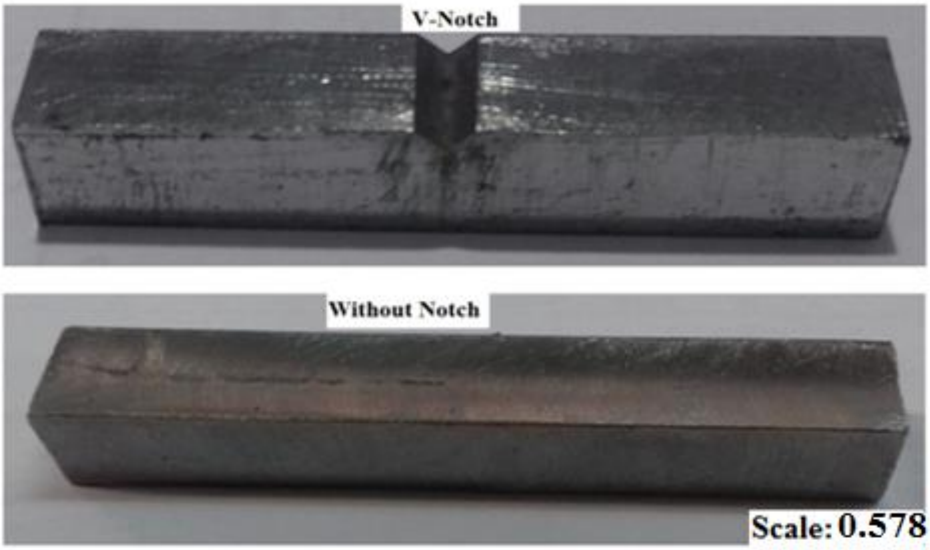


Dimensions	
Length (L) [mm]	80±2
Width (b) [mm]	10.0±0.2
Thickness (h) [mm]	4.0±0.2

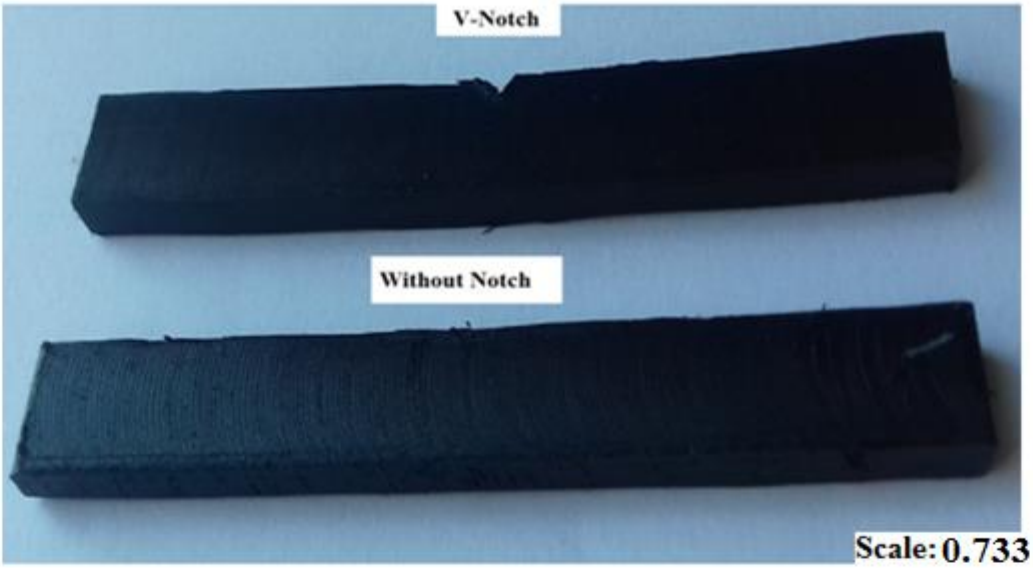
Figure 3.4: The standard Charpy test specimen of HDPE

3.2.2 Charpy Test Procedure

Figure 3.5 shows the specimens of API 5L X60 and HDPE prepared in this work. In the present, tests specimens of API 5L X60 and HDPE were examined in Libyan Iron and Steel company's laboratory and Mechanical Engineering laboratory at Near East University, respectively. The Charpy impact machine is presented in Figure 3.6

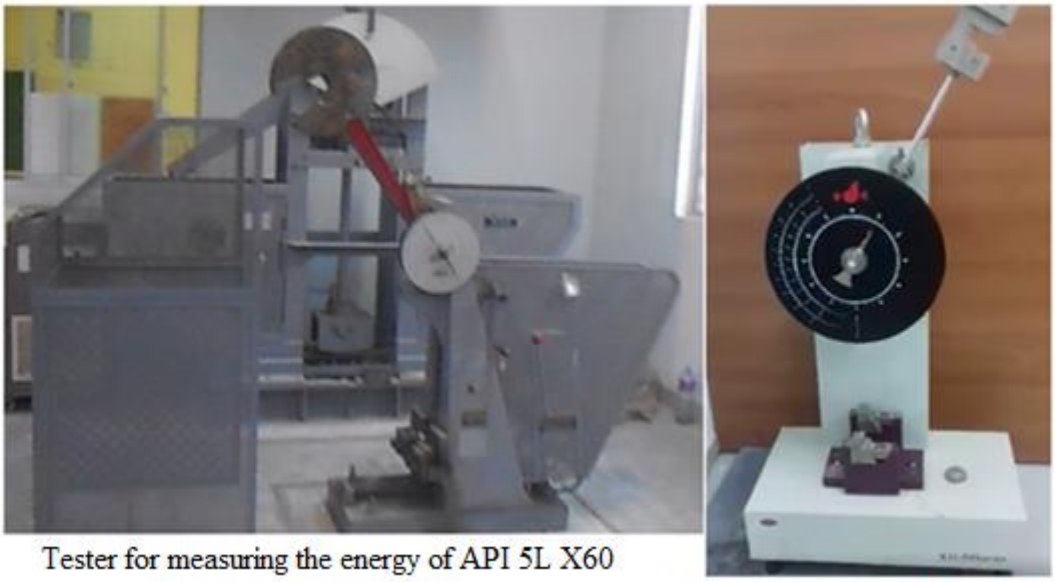


(a) API 5L X60



(b) HDPE

Figure 3.5: Specimens of API 5L X60 steel and HDPE



Tester for measuring the energy of API 5L X60 (Libyan Iron Steel works)

Charpy tester for HDPE (NEU)

Figure 3.6: Charpy impact test

Generally, three tests were performed for API 5L X60 and HDPE at various temperatures. An overview of the tests performed can be seen in Table 3.4.

Table 3.4: Charpy test performed

Material	Flaw geometry	Temperature [℃]	No. tests performed
API 5L X60	V-Notch	25℃ and -196℃	3
	Without Notch	25℃ and -196℃	3
HDPE	V-Notch	20℃	3
	Without Notch	20℃	3

3.2.2.1 API 5L X60

In this part, the CI-No 30 pendulum (hammer) type with capacity of 30 kg-m was used for testing the impact of API 5L X60 according ASTM E23-00 (Appendix 3).

The specimens can be divided into two group before testing in the Charpy impact test as follows

1. Three specimens of API 5L X60 was tested at room temperature (25℃).
2. Three specimens of API 5L X60 was immersed in nitrogen liquid before testing them.

The three tests specimens of API 5L X60 were cooled down in a bath containing nitrogen liquid for tests performed at temperatures -196℃. The specimens cooled down in the Nitrogen bath were immersed in the liquid for 30, 90 and 180 minutes. After sufficient cooling, the specimens were inserted directly into the test machine and tested. For the test, a hammer strokes the notched specimens then the absorbed energy by each specimen was recorded. The tests were performed and energy was recorded using standard Charpy impact machine. Three specimens were tested in each step and the average values were considered.

The measured total energy, E, the energy given by the instrumented Charpy instrument, and the measuring angle, β, of each test were recorded. The measured E values of API 5L X60 can be calculated using equations 3.1 and 3.2, respectively.

$$\mathrm{E} = \mathrm{Energy}\ (\mathrm{kg.f.m}) \times 9.80665 \tag{3.1}$$

$$\mathrm{E} = \mathrm{Pd}(\cos\beta - \cos\alpha) \tag{3.2}$$

where

Pd: Torque of the hammer

α: Starting angle of the hammer before impact (α = 143°)

β: Angle after impacting the specimen

3.2.2.2 HDPE at NEU

Charpy impact tester XJJ-50 in the mechanical Laboratory of Near East University was used to test the HDPE material according BS EN ISO 179-1:2001 (Appendix 2). Three specimen samples were tested at 20℃ and relative humidity of 65%. For the test, a hammer strokes the notched specimens then the absorbed energy by each specimen was recorded.

The tests were performed and energy was recorded using standard Charpy impact machine. Three specimens were tested in each step and the average values were considered.
The measured total energy, E, the energy given by the instrumented Charpy instrument, and the measuring angle, β, of each test were recorded. The measured E values of HDPE can be calculated using Equation 3.3.

$$E = Pd(\cos\beta - \cos\alpha) \tag{3.3}$$

where;
Pd: Torque of the hammer 8.03878 N.m
α: Starting angle of the hammer before impact (α = 150°)
β: Angle after impacting the specimen

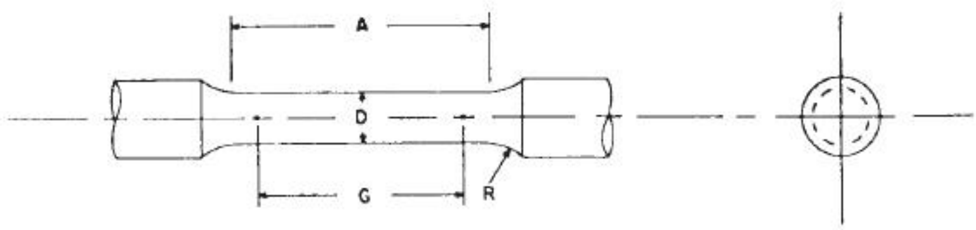
3.3 Tensile testing

The tension test is one of the most commonly used tests for evaluating materials. In its simplest form, the tension test is accomplished by gripping opposite ends of a test specimen within the load frame of a test machine; A tensile force is applied by the machine, resulting in the gradual elongation and eventual fracture of the test specimen. During this process, force extension data, a quantitative measure of how the test specimens test provides force extension data that can quantify several important mechanical properties of a material. These mechanical properties determined from tensile tests include the following:

- Young's modulus
- Yield strength
- Ultimate tensile strength
- Elongation

3.3.1 Specimen Preparation of API 5L X60 Pipeline Steel

Tests were conducted in accordance with ASTM E 8M – 04 standard (Appendix 4). Test specimens were cut-out of the steel pipe samples and prepared with the dimensions shown in Figure 3.7. The diameter of the specimen used in this work was 9 mm. The tensile tests were conducted firstly at room temperature (25℃). Then, the test specimens were cooled down in a bath containing nitrogen liquid for tests performed at temperatures -196℃. The specimens cooled down in the nitrogen bath were immersed in the liquid for 90 minutes. After 90 min, the specimens were inserted directly into the test machine and tested.



	Dimensions , mm	
	Standard specimen	**Size specimen proportional to standard**
	12.5	9
G-Gage length	62.5±0.1	45.0±0.1
D-Diameter (Note 1)	12.5±0.2	9.0±0.1
R-Radius of fillet, mm	10	8
A-Length of reduced section, mm (Note 2)	75	54

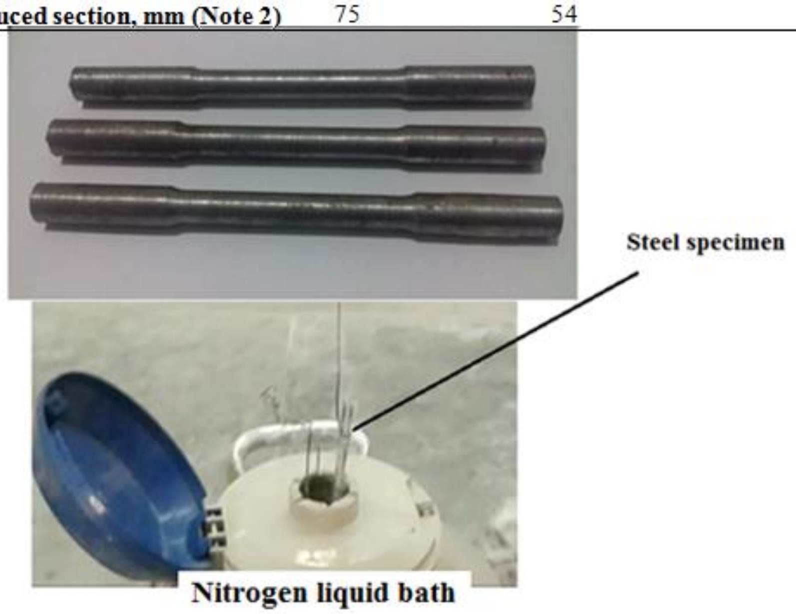


Figure 3.7: Measurements and tolerances of tensile stress test specimens, machined according to standard (Designation: E 8M – 04)

3.3.2 Tensile Testing Procedure of API 5L X60 Pipeline Steel

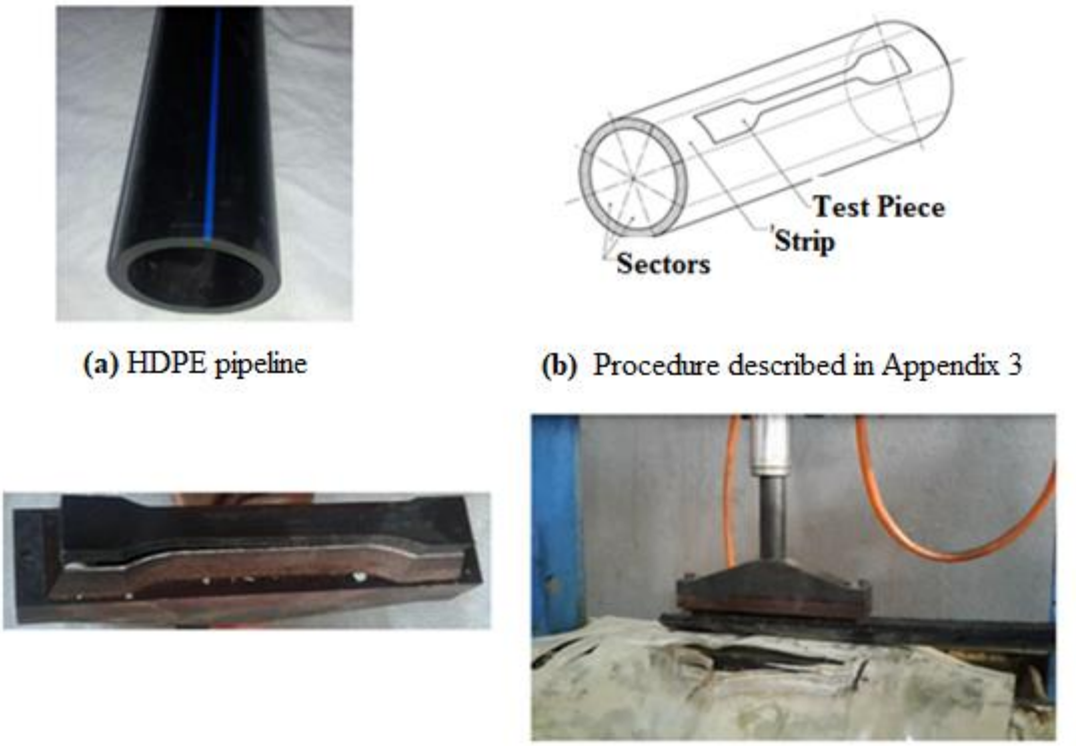
Tensile tests apply forces directly the material sample, usually by using clamps to securely grip two opposite ends of the sample and then pulling the ends away from each other. As the force is slowly increased the stress on the material slowly increases and the sample elongates until it reaches its maximum strain and the material breaks. Tensile tests were conducted at various temperatures using universal tensile tester machine and EZ tensile machine shown in Figure 3.8 and 3.9, respectively. Ultimate load [kg], Ultimate tensile stress [kPa], and elongation at yield, El, (%) were recorded. Three specimens were tested in each step and the average values were considered.

Figure 3.8: Universal tensile testing machine at Libyan steel and Iron Company of Misurata/Libya

Figure 3.9: LLOYD EZ 50-universal tensile testing machine at the Mechanical Engineering Laboratory, NEU

3.3.3 Preparation of HDPE Specimens

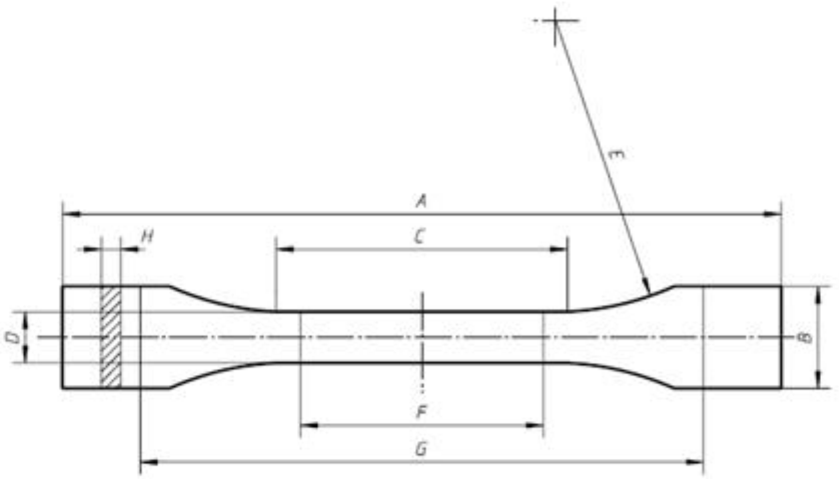
The cutting method for HDPE specimens is described in ISO 6259-1 (see Appendix 5). Cut strips from the pipe as supplied, i.e. which has not been heated or flattened, so that their axis is parallel to the axis of the pipe and the positions from which the strips are taken conform to pipes of nominal outside diameter greater than 63 (Appendix 6). Cut strips from the length in such a way that they are equally distributed around the circumference of the pipe as shown in Figure 3.10. The procedure of cutting method for HDPE specimens was occurred at room temperature (Appendix 3). Test specimens were cut-out of the HDPE pipe samples and prepared with the dimensions shown in Figure 3.11 in accordance with ISO 6259-3. The tensile tests were conducted firstly at room temperature (25°C). Then, the test specimens were cooled down in a bath containing nitrogen liquid for tests performed at temperatures -196°C. The specimens cooled down in the Nitrogen bath were immersed in the liquid for 90 minutes. After 90 min, the specimens were inserted directly into the test machine and tested.



(a) HDPE pipeline (b) Procedure described in Appendix 3

(c) Stamp (d) Stamp sample and hydrulic press pipeline

Figure 3.10: Preparation of test sample from HDPE pipeline material



Symbol	Description	Dimensions mm
A	Overall length (min.)	150
B	Width of ends	20 ± 0,2
C	Length of narrow, parallel-sided portion	60 ± 0,5
D	Width of narrow, parallel-sided portion	10 ± 0,2
E	Radius	60
F	Gauge length	50 ± 0,5
G	Initial distance between grips	115 ± 0,5
H	Thickness	That of the pipe

(a) Tensiletest dimensions (Appendix 3)

(b) Simple tested at room temperature

(c) Sample tested in liquid nitrogen

Figure 3.11: Measurements and tolerances of tensile stress test specimens of HDPE machined according to ISO 6259-3 standard (Appendix 3)

3.3.4 Tensile Testing Procedure of HDPE

Tensile tests were conducted at various temperatures using Niversal tensile tester machine and EZ tensile machine as shown in Figure 3.9 and 3.12. Ultimate load [kg], Ultimate tensile stress [kPa], and elongation at yield, El, (%) were also recorded. Three specimens were tested in each step then the average values were considered at each temperature.

Figure 3.12: Universal tensile tester at Misurata Factory, Libya

3.4 Rockwell Hardness Testing

The hardness of steel specimens was tested according to ASTM E 18-00 (Appendix 7), using the Rockwell Hardness testing machine for API 5L X60 and Shore hardness tester for HDPE at the Libyan of Iron and Steel company workshop as shown in Figure 3.9 and Figure 3.10, respectively.

This test machine is the widely accepted due to its speed, freedom from personal errors, ability to distinguish small hardness difference, and a small size of indentation. The hardness is measured according to the depth of indentation, under a constant load. In order to do the Rockwell Test the following procedures must be followed:

- Position the specimen to be tested close to the indenter.
- Apply the minor load to establish a zero reference position.
- Apply the major load for a specified time period called a dwell time, in this case 60seconds.
- Release the major load leaving the minor load applied.

The Rockwell number represents the difference in depth from the zero reference position as a result of the applied major load. Three specimens (Figure 3.11) were tested in each step and the average values were considered.

The hardness testier was conducted firstly at room temperature (25℃). Then, the test specimens (see Figure 3.12) were cooled down in a bath containing Nitrogen liquid for tests performed at temperatures -196℃. The specimens cooled down in the Nitrogen bath were immersed in the liquid for 90 minutes. After 90 min, the specimens were inserted directly into the test machine and tested.

Figure 3.13: Rockwell Hardness tester (B) of the Libyan Iron and Steel Company, Misurata Factory, Libya

Figure 3.14: Shore hardness tester of Libyan Iron and Steel Company, Misurata Factory, Libya

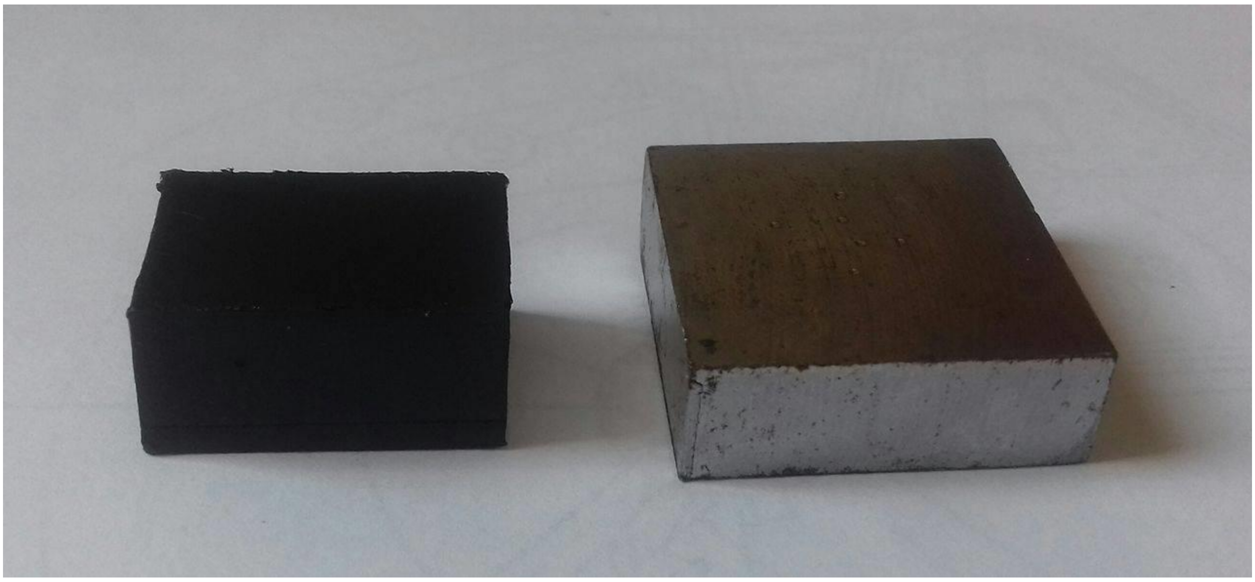


Figure 3.15: Hardness specimens of HDPE and API 5LX60

Figure 3.16: Nitrogen liquid bath for cooling the hardness specimens

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Charpy Impact Test Behaviors

4.1.1 API 5L X60 steel

The results are seen in the last two columns of Table 4.1. It can be noted that the Charpy absorbed energy values for the un-notched API 5L X60 samples did not change much at room and liquid nitrogen temperatures, Table 4.1 and Table 4.2. On the other side, for the V-notched samples the Charpy absorbed energy values dropped from approximately 210J to 5J once cooled down from RT to NT. Gotefroid et al. (2014) reported the Charpy absorbed energy was 169 J at RT for V-notched API 5L X60 steel.

The photographs of the test specimens can be seen in Figure 4.1 and 4.5. It can be seen that the un-notched samples didn't fracture neither at RT or Nt. However, notched samples fractured both at RT and NT. Hence the steel was sensitive to notches at both temperatures. As seen n Figure 4.2, the notched samples broke in a ductile manner at room temperature. However, they broke in a brittle manner when they were cooled in liquid Nitrogen. On the other side, it is seen in Figure 4.1 that the un-notched samples kept their ductility not only at RT but also at liquid nitrogen temperature. Hence, the notch and crack free API 5L X60 steel pipelines can remain safe at sub-zero temperatures.

Table 4.1: Data from the Charpy impact tests of API 5LX60 steel samples at RT (25℃)

No. of test	V-Notch	Without Notch	V-Notch	Without Notch	V-Notch	Without Notch
	β[°]		**Energy [kgf.m]**		**Absorbed Energy [J]**	
specimen 1	58.50	2.00	21.865	30.00	214.422	294.20
specimen 2	62.00	2.50	20.970	30.02	205.645	296.16
specimen 3	60.00	2.50	21.490	30.02	210.745	296.16
Average	60.16	2.33	21.442	30.13	210.271	295.51

Table 4.2: Data from the Charpy impact tests of API 5LX60 steel cooled in Liquid Nitrogen

Time	30 min					
No. of test	V-Notch	Without Notch	V-Notch	Without Notch	V-Notch	Without Notch
	β [°]		Energy [kgf.m]		Absorbed Energy [J]	
specimen 1	130.000	6.00	2.680	29.920	26.282	293.415
specimen 2	130.500	6.50	2.570	29.900	25.203	293.219
specimen 3	130.000	5.50	2.680	29.950	26.282	293.709
average	130.167	6.00	2.625	29.925	25.922	293.448
Time	**90 min**					
No. of test	**V-Notch**	**Without Notch**	**V-Notch**	**Without Notch**	**V-Notch**	**Without Notch**
	β [°]		**Energy [kgf.m]**		**Absorbed Energy [J]**	
specimen 1	140.500	9.50	0.555	29.765	5.443	291.895
specimen 2	140.500	10.00	0.555	29.740	5.443	291.650
specimen 3	141.000	11.50	0.460	29.650	4.511	290.767
average	140.667	10.33	0.523	29.718	5.132	291.437
Time	**180 min**					
No. of test	**V-Notch**	**Without Notch**	**V-Notch**	**Without Notch**	**V-Notch**	**Without Notch**
	β [°]		**Energy [kgf.m]**		**Absorbed Energy [J]**	
specimen 1	141.000	29.00	0.440	28.820	4.315	282.628
specimen 2	141.500	30.00	0.535	27.680	5.247	271.448
specimen 3	141.500	32.00	0.535	27.370	5.247	268.408
average	141.333	30.33	0.535	27.525	4.936	274.161

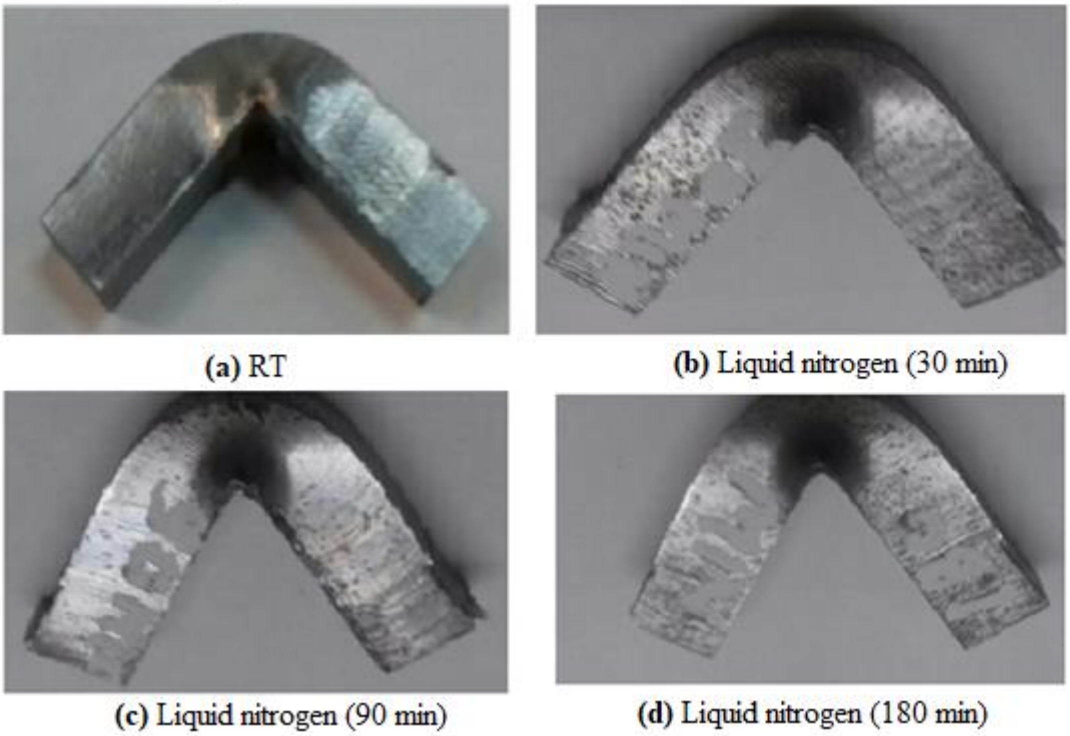


Figure 4.1: Testing specimens of API 5L X60 (without Notch) after impact test at various temperature (10mm (width)× 10mm (thickness)× 55mm(length))

Figure 4.2: Testing specimens of API 5L X60 (with Notch) after impact test at various temperature (10mm (width)× 10mm (thickness)× 55mm(length)) with 2mm depth (V)

4.1.2 HDPE

Notched and un-notched HDPE samples were tested at 20℃ and 65% relative humidity in the mechanical Engineering Laboratory of Near East University (NEU).

The un-notched samples did not fracture (Figure 4.3) whereas notched sample fractured (Figure 4.4). The test data and the Charpy absorbed energies were found 122 kJ/m^2 and 44 kJ/m^2 for the un-notched and notched samples, respectively. HDPE specimens were not tested at liquid Nitrogen temperature similar data can be found in the literature (ISO Industries, 2000). Therefore, the room temperature behaviour of the HDPE samples were similar to that of API 5L X60 steel samples. HDPE was sensitive to notches.

Figure 4.3: HDPE samples without notch after impact testing at RT carried out in Mechanical Engineering Laboratory, NEU (10mm (width)× 4mm (thickness)× 80mm (length))

Figure 4.4: HDPE samples with notch after impact testing at RT carried out in Mechanical Engineering Laboratory, NEU) (10mm (width)× 4mm (thickness)× 80mm(length)) with 2mm depth (V)

Table 4.3: Data from Charpy impact tests of HDPE samples with and without notches

	No. of test	**β[°]**	**Absorbed Energy [J]**	**a [kJ/m^2]**
With notch	1	135	1.28	40.0
	2	136	1.18	35.9
	3	130	1.89	56.1
	Average			44.0
Without notch	1	113	3.82	95.5
	2	93	6.74	163.5
	3	109	4.35	106.0
	Average			121.7

4.2 Tensile Test Behaviors

The photographs of the tension test samples of API 5L X60 steel and HDPE samples are given in Figures 4.5 and 4.6, respectively. The tension test data for the two material are given in Table 4.4 and 4.5.

It's seen that in all samples the fracture occurred within the gauge length. In the case of API 5L X60 steel varied a little whither the sample was tested at room temperature or liquid Nitrogen temperature. The sample soaked for 180 minutes in the liquid Nitrogen elongation about 10% more than its counterpart tested at room temperature.

An increase of 17% in ultimate tensile strength was noted. It appeared that cryogenic heat treatment had a positive effect both on tensile strength and fracture elongation of API 5L X60 steel samples.

No specification is given for stress at yield for the HDPE pipe material in the ISO 6259-3:1997 standard. However, it is recommended that the mean elongation at yield must be greater that 350%. The elongation in this study was measured as 369% at room temperature.

The tensile strength at yield and break were 470 kPa and 440 kPa, respectively as seen in Table 4.5. Sharp decreases was observed both in elongation and also in strength when the HDPE samples were cooled in Liquid nitrogen before tests. For instance, the tensile strength at yield was 150kPa and elongation was 33% when the sample was cooked for 180min in the elongation curves of HDPE samples can be seen in Figure 4.7.

As in the case of un-notched Charpy impact test samples, the tensile behaviour of API 5L X60 steel samples were unchanged when cooled in liquid Nitrogen, Figure 4.5. The tension test sample were machined following the ASTM E8 standard and were free at any notches and other surface defects. Unlikely, the tension test samples of HDPE lost their ductility when cooled in liquid Nitrogen, Figure 4.6. The HDPE may not be a suitable material for pipelines that would operate at sub-zero temperatures.

(a) RT **(b)** Liquid nitrogen (30 min) **(c)** Liquid nitrogen (90 min) **(d)** Liquid nitrogen (180 min)

Figure 4.5: Tensile test specimens of API 5L X60 steel fractured at different temperature at Libyan iron and Steel Company, Misurata Libya (size specimens of 9mm and A-length of reduced section of 54mm)

(a) RT

(b) Liquid nitrogen (30 min) (c) Liquid nitrogen (90 min) (d) Liquid nitrogen (180 min)

Figure 4.6: Tensile test specimens of HDPE fractured at different temperatures at Libyan iron and Steel Company, Misurata Libya (overall length is 150mm and length of narrow parallel -sided portion is 60$\pm$0.5mm)

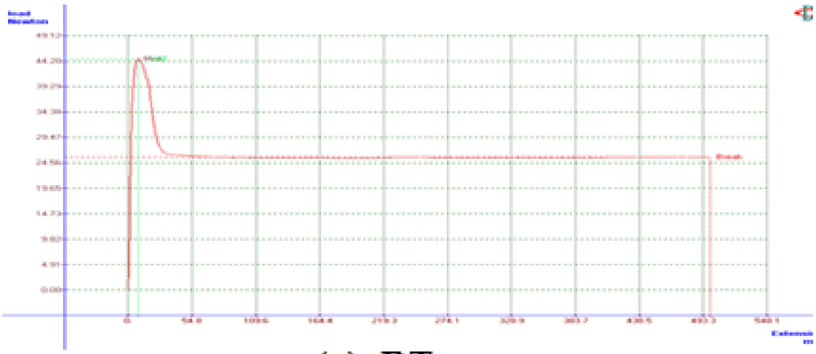
Table 4.4: Data from tension test samples of API 5L X60 steel fractured at various temperatures

No. of test	Area [mm^2]	Diameter [mm]	Gauge length [mm]	Ultimate load [kN]	Ultimate tensile stress [MPa]	New length [mm]	tensile strain [mm/mm]	El [%]
				RT				
specimen 1	63.60	9.00	45.00	3.08	484.3	54.00	0.20	20.00
specimen 2	63.60	9.00	45.00	3.13	492.1	54.00	0.20	20.00
specimen 3	63.60	9.00	45.00	3.30	518.9	54.00	0.20	20.00
				NT				
Time [min]					**30 min**			
specimen 1	63.60	9.00	45.00	3.50	550.3	54.30	0.21	20.67
specimen 2	63.60	9.00	45.00	3.51	551.9	54.50	0.21	21.11
specimen 3	63.60	9.00	45.00	3.53	555.0	54.20	0.20	20.44
Time [min]					**90 min**			
specimen 1	63.60	9.00	45.00	3.36	528.3	54.77	0.22	21.71
specimen 2	63.60	9.00	45.00	3.79	595.9	54.77	0.22	21.71
specimen 3	63.60	9.00	45.00	3.70	581.8	54.76	0.22	21.69
Time [min]					**180 min**			
specimen 1	63.60	9.00	45.00	3.90	613.2	55.10	0.22	22.44
specimen 2	63.60	9.00	45.00	3.88	610.1	55.00	0.22	22.22
specimen 3	63.60	9.00	45.00	3.87	608.5	54.90	0.22	22.00

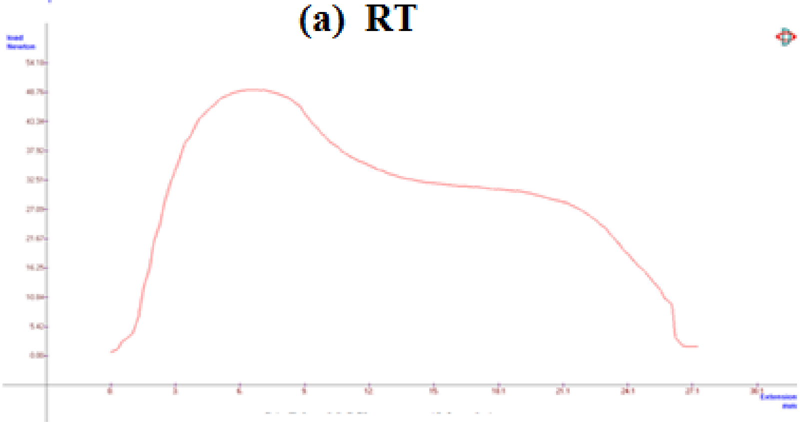
Table 4.5: Data from tension test samples of HDPE fractured at various temperatures

	Area [mm^2]	Yield Force [N]	Yield Elongation [mm]	Break Force [kN]	Break Elongation [mm]	Tensile Strength at Yield [kPa]	Tensile Strength at Break [kPa]	Elongation [%]
				RT				
specimen 1	100	46.5	11.62	26.7	400.06	470	440	368.97
				NT				
Time [min]					**30 min**			
specimen 1	100	46.43	7.58	28.63	31.4	270	250	65.6
Time [min]					**90 min**			
specimen 1	100	52.7	15.12	28.22	29.3	170	140	41.53
Time [min]					**180 min**			
specimen 1	100	55.13	17.52	28.33	21.9	150	130	32.75

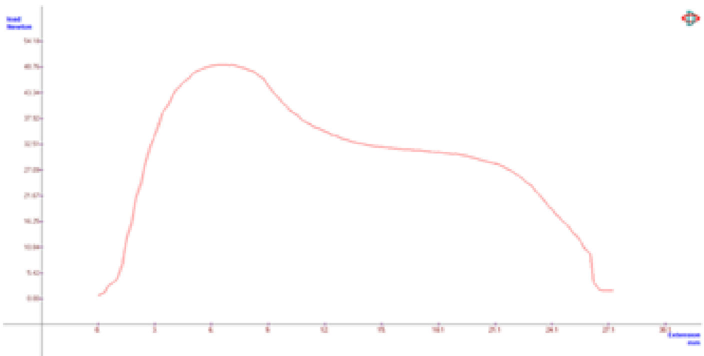
The yield force, ultimate tensile stress and EI values for HDPE at RT and NT obtained from these tests are given in Figure 4.7 (summarized in Table 4.5).



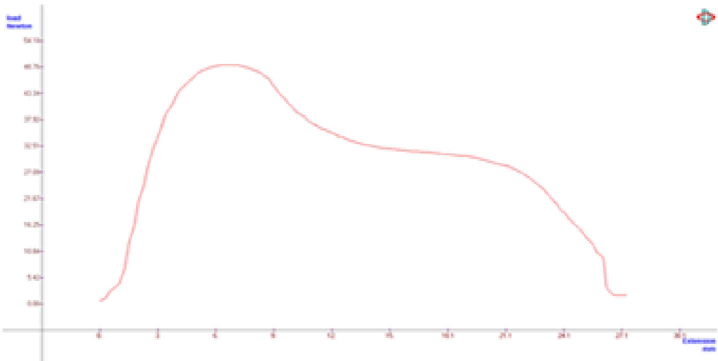
(a) RT



(b) Liquid Nitrogen (30 min)



(c) Liquid Nitrogen (90 min)



(d) Liquid Nitrogen (180 min)

Figure 4.7: Load vs elongation curves of HDPE samples fractured at various temperatures

4.3 Hardness Test

The hardness test data for the API 5L X60 steel and HDPE are given in Table 4.6 and Figure 4.8. Increases in HRC values are found for API 5L X60 steel samples when cooled in liquid Nitrogen. On the contrary, a small decrease in the shore hardness values of the HDPE samples were noted. The results of this study for API 5L X60 steel compared with those of Godefriod et al., (2014) in Table 4.6. It can been seen that the mechanical properties of API 5L X60 steel found in this work are in agreement with that of Godefriod et al., (2014).

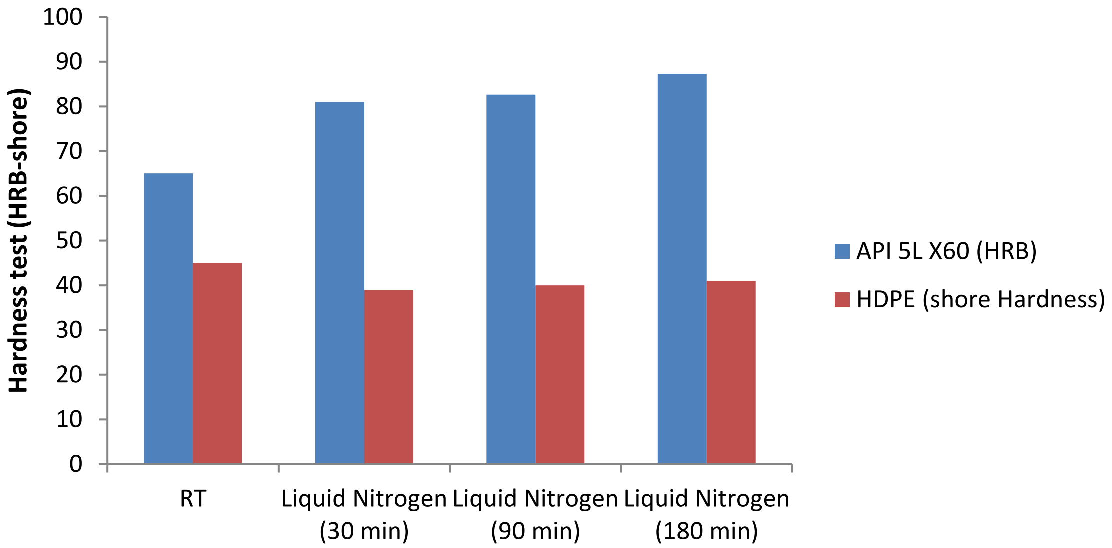


Figure 4.8: Hardness values of API 5L X60 steel (HRB) and HDPE (Shore hardness) samples

Table 4.6: Hardness Testing for API 5L X60 and HDPE samples

Temperature	API 5L X60 (HRB)	HDPE (shore Hardness)
RT	65	45
Liquid Nitrogen (30 min)	81	39
Liquid Nitrogen (90 min)	82.66	40
Liquid Nitrogen (180 min)	87.3	41

Table 4.6: Mechanical properties of API 5L X60 at room temperature

Property	Current Study	Previous study (Godefroid et al. (2014))
Ultimate tensile stress [MPa]	498.3	576
El [%]	20	14
Rockwell hardness B	65	59
Charpy impact test [J]	210	169

CHAPTER 5
CONCLUSIONS

5.1 Conclusions

The tensile and Charpy impact tests of the oil pipeline API 5L X60 were carried out both at RT (Room Temperature) and also at liquid NT (Liquid Nitrogen Temperature) following either ASTM or EN ISO standards. Moreover, the effect of RT and NT on the properties of steel API 5L X60 were investigated. The same procedure was also followed to characterize the same properties of a different pipeline material HDPE (High Density Polyethylene) particularly, used to transport water and natural gas.

The results showed that the Charpy impact energy of the un-notched API 5L X60 steel samples did not change much when cooled from room temperature to liquid Nitrogen temperature. On the contrary, the Charpy impact energy of notched specimen was reduced sharply from 210J to 5J once cooled from room temperature to liquid temperature. The tensile strength fracture elongation and hardness were all increased when steel sample was cooled in liquid Nitrogen.

The Charpy impact energies were found as 122 kJ/m^2 to 44 kJ/m^2 for the un-notched and notched HDPE samples tested at room temperature. The samples were not tested at liquid nitrogen temperature. Unlike the API 5L X60 steel, the tensile strength and fracture elongation reduced significantly when cooled down to liquid nitrogen temperature. A small decrease was also noted in its hardness.

It is understood that the un-notched API 5L X60 steel sample did not suffer a ductile to brittle transition when cooled down to liquid Nitrogen temperature. However, this was not the case for the HDPE samples, Hence, pipelines made of API 5L X60 can be used at sub-zero temperatures, but the HDPE is not a suitable material for sub-zero temperature use.

5.2 Recommendations for Future Work

i. Further mechanical and metallurgical tests can be carried out on both pipeline materials. Fatigue tests and examination of fracture surfaces are needed for a more detailed research.

ii. A similar study can be made on the other alternative pipeline materials. Then, a comparison can be made for specific needs in the pipeline materials.
iii. In this work, the material used was API 5L X60 steel. The experimentation can be also be done with other material such as composites material to see the effect of the parameters on the mechanical properties and microstructure can be checked.

REFERENCES

Ali, B., Abdullah, S., Nuawi, M., & Ariffin, A. (2013). Correlation of absorbed impact with calculated strain energy using an instrumented Charpy impact test. *Indian Journal of Engineering and Materials Sciences*, *20*, 504-514.

Askeland, D. (1990). *The science and engineering of materials*. Chapman and Hall.

Askeland, D., Fulay, P., & Wright, W. (2006). *The Science and Engineering of Materials* (6th ed.). USA: Cengage Learning.

AWWA. (2006). *PE Pipe - Design and Installation - Manual of Water Supply Practices, M55 (1st ed.)*. American Water Works Association / Colorado.

Bannantine, J. A., Comer, J. J., & Handrock, J. L. (1990). *Fundamentals of metal fatigue analysis*. Englewood Cliffs, NJ: Prentice Hall.

BS 2782-3:Methods. (1976). Mechanical properties - Methods 320A to 320F tensile strength, elongation and elastic modulus, Methods for testing plastics, BS 2782 part 3.

Coulton, J. P. (1996). *Effect of elevated strain rates on the mechanical performance of polyethylene structures* (Doctoral dissertation, University of Warwick).

Darvell, B. W. (2009). *Materials Science for Dentistry* (9th ed.). CRC Press.

Davis, J. R. (1998). *ASM Specialty Handbook: Tool Materials*. Material Parks, OH: ASM International.

Florén, M. (2011). *Test Method for Performance Evaluation of Abrasive Waterjet Cutting*(Master's thesis, Blekinge Institute of Technology, Sweden).

Gambhir, M. L., & Jamwal, N. (2014). *Building and construction materials: Testing and quality control : lab manual*. Tata McGraw-Hill Education

Godefroid, L. B., Cândido, L. C., Toffolo, R. V., & Barbosa, L. H. (2014). Microstructure and mechanical properties of two Api steels for iron ore pipelines. *Materials Research*, 17, 114-120.

Gupta, K. (2015). *Engineering Materials: Research, Applications and Advances*. USA: CRC press.

Heness, G., & Cortie, M. B. (2012). *Material science and engineering*. Frenchs Forest, N.S.W.: Pearson Education Australia.

Herrman, K. (2011). *Hardness Testing: Principles and Applications*. ASM International.

Hoechst, C. (1991). *Designing with Plastic: The Fundamentals*. USA.

ISCO Industries. (2000). Total Piping Solutions. Retrieved April 30, 2018, from http://www.isco-pipe.com/home/

Javaherdashti, C.I., Henry Tan, Corrosion and Materials in the Oil and Gas Industries 2013: Taylor & Francis Group

Javaherdashti, R., & Tan, H. (2013). *Corrosion and materials in the oil and gas industries*. Taylor & Francis Group.

Koerner,R.M.(2012).DesigningwithGeosynthetics,6thEdition,Vol.1,PrenticeHall.

Koo, J. Y., Luton, M., Bangan, M., & Petkovi, R. (2004). Metallurgical Design of Ultra High-strength Steels For Gas Pipelines. *International Journal of Offshore and Polar Engineering*, 14, 9-20.

Laryee. (2018). Izod And Charpy Universal Impact Testing Machine. Retrieved January 17, 2018, from http://www.laryee.com/impact/nometal-impact-TM/51.htm

Mishra, D. (2014). *Thermo-mechanical Processing of API-X60 Grade Pipe Line Steel* (Master's thesis, National Institute of Technology).

Müller, D. (1982). *Review of mathematical design methods for thermoplastic machine parts*. BASF Atiengesellschaft Ludwigshafen Germany.

Nicholas, T. (1981). Tensile Testing of Materials at High Rates of Strain. *Experimental Mechanics*, *21*, 177-185.

Öjmertz, C. (2006). *A guide to waterjet cutting*. Ronneby: Water Jet Sweden.

Plastics Pipe Institute (2008). *The Plastics Pipe Institute Handbook of Polyethylene Pipe* (2nd Edition). USA

Singh, N. (2009). *Experimental study and parametric design of impact testing methodology* (Master's thesis, Thapar university).

Sunny-steel. (2011). Alloy Pipe, Seamless tubes, Welded pipe, Pipe fittings. Retrieved April 30, 2018, from http://www.sunnysteel.com/

Tóth, L., Rossmanith, H., & Siewert, T. (2002). Historical background and development of the Charpy test. *European Structural Integrity Society*, *30*, 3-19.

Verhoeven, J.D. (2007). *Steel Metallurgy for the Non-Metallurgist*. Materials Park, OH, USA: ASM International.

Watkins, R. K. (2004). Pipe and Soil Mechanics for Buried Corrugated HDPE Pipe. *Pipeline Engineering and Construction*, *2*, 1-4.

Westmoreland Mechanical Testing & Research. (2002). Charpy Impact Test: ASTM E23. Retrieved November 14, 2016, from http://www.wmtr.com/en.charpy.html

Westmoreland Mechanical Testing and Research, WMTR,. (2018). Charpy Impact Test: ASTM E23. Retrieved January 2, 2018, from http://www.wmtr.com/en.charpy.html l

Wright, W., & Askeland, D. (2016). *The Science and Engineering of Materials* (7th ed.). USA: CENGAGE Learning.

APPENDICES

APPENDIX 1

CHEMICAL ANALYSIS OF API 5L X60 steel

Table 1. Chemical composition of API 5L X60 at room temperature (25℃)

	Element	Fe	C	Mn	Si	P	S	Ni	Ci	Cu	Mo
	Units	%	%	%	%	%	%	%	%	%	%
Number	1	98.64216	0.15357	0.89711	0.18281	0.01449	0.00363	0.01785	0.02216	0.01781	0.00052
	2	98.66088	0.15285	0.88419	0.17987	0.01408	0.00359	0.01792	0.02192	0.01769	0.0053
	3	98.66805	0.1503	0.88184	0.17822	0.01393	0.00352	0.01784	0.02187	0.01792	0.0057
	AVG	98.65703	0.15224	0.887713	0.1803	0.014167	0.00358	0.01787	0.021983	0.017807	0.00384
	SD	0.013366	0.001816	0.008219	0.002329	0.000288	0.000055	0.000042	0.000154	0.000118	0.000024
	SD%	0.01	1.19	0.93	1.29	2.04	1.53	0.23	0.7	0.66	4.38

	Element	V	Ti	Al	Nb	W	As	Sn	Co	Pb	B
	Units	%	%	%	%	%	%	%	%	%	%
Number	1	0.00317	0.00207	0.04258	0.00264	0.00039	0.00171	0.00398	0.00344	0.00209	0.00013
	2	0.00299	0.00194	0.04223	0.00256	0.00036	0.00175	0.00376	0.00343	0.00185	0.00015
	3	0.00291	0.00189	0.04188	0.00256	0.000063	0.00178	0.00387	0.00341	0.00178	0.00014
	AVG	0.003023	0.0019667	0.04223	0.002587	0.000271	0.001747	0.00387	0.003427	0.001907	0.00014
	SD	0.000136	0.000093	0.000093	0.000044	0.00015	0.000037	0.000109	0.000013	0.000164	0.000007
	SD%	4.48	4.71	4.71	1.68	32.65	2.1	2.81	0.39	8.59	4.78

	Element	Sb	Ca	Mg	Zn	Ce	N
	Units	%	%	%	%	%	%
Number	1	0.00055	-0.00004	0.00011	0.00145	0.00219	-0.01858
	2	0.00067	0.00002	0.00012	0.00141	0.00144	-0.0182
	3	0.0007	-0.00015	0.00011	0.00141	0.00128	-0.01811
	AVG	0.00064	-0.00006	0.000113	0.001423	0.001637	-0.0183
	SD	0.000082	0.00009	0.000004	0.000027	0.000484	0.000248
	SD%	12.75	161.98	3.08	1.92	29.61	1.36

Table 2. Chemical composition of API 5L X60 steel cooled in Liquid Nitrogen for 30min

	Element	**Fe**	**C**	**Mn**	**Si**	**P**	**S**	**Ni**	**Ci**	**Cu**	**Mo**
	Units	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**
Number	**1**	98.71439	0.14326	0.84746	0.16841	0.0107	0.00316	0.01658	0.02158	0.01623	0.0011
	2	98.727	0.13341	0.85412	0.16753	0.01163	0.00327	0.01677	0.02167	0.0161	0.00105
	3	98.727	0.13266	0.85223	0.16005	0.01135	0.00299	0.01654	0.02162	0.01589	0.00116
	AVG	**98.7228**	**0.1364433**	**0.85127**	**0.16533**	**0.011227**	**0.00314**	**0.01663**	**0.021623**	**0.016073**	**0.001103**
	SD	**0.007017**	**0.005912**	**0.003433**	**0.000968**	**0.000478**	**0.000142**	**0.000124**	**0.000047**	**0.000174**	**0.000055**
	SD%	**0.01**	**4033**	**0.4**	**0.58**	**4.26**	**4.52**	**0.74**	**0.22**	**1.08**	**5.02**

	Element	**V**	**Ti**	**Al**	**Nb**	**W**	**As**	**Sn**	**Co**	**Pb**	**B**
	Units	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**
Numbe r	**1**	0.00377	0.00208	0.0414	0.00349	0.00202	0.00166	0.00358	0.00335	0.00393	0.00018
	2	0.00364	0.00204	0.03993	0.00328	0.00113	0.00164	0.00358	0.00331	0.00376	0.00015
	3	0.00357	0.002	0.04002	0.00318	0.00108	0.00182	0.00358	0.00326	0.00358	0.00017
	AVG	**0.00366**	**0.00204**	**0.04045**	**0.003317**	**0.00141**	**0.001707**	**0.00358**	**0.003307**	**0.003757**	**0.000167**
	SD	**0.000101**	**0.000039**	**0.000821**	**0.000156**	**0.000536**	**0.000096**	**0.000131**	**0.000037**	**0.000175**	**0.000012**
	SD%	**2.76**	**1.89**	**2.03**	**4.71**	**38.92**	**5.64**	**3.61**	**1.12**	**4.65**	**7.24**

	Element	**Sb**	**Ca**	**Mg**	**Zn**	**Ce**	**N**
	Units	**%**	**%**	**%**	**%**	**%**	**%**
Numbe r	**1**	0.00061	0.00244	0.00015	0.0015	0.00316	0.01666
	2	0.00053	0.00019	0.0001	0.00137	0.00291	0.01682
	3	0.00031	0	0.00011	0.00139	0.00255	0.01668
	AVG	**0.000483**	**0.00088**	**0.00012**	**0.00142**	**0.002873**	**0.01672**
	SD	**0.000154**	**0.001466**	**0.000029**	**0.000073**	**0.000311**	**0.000088**
	SD%	**31.9**	**194.84**	**24.01**	**5.16**	**10.81**	**0.53**