

**MECHANICAL PROPERTIES ANALYSIS OF
ADBLUE TANK MATERIAL**

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**ADBLUE TANK MALZEMESİNİN MEKANİK
ÖZELLİKLERİNİN İNCELENMESİ**

YÜKSEK LİSANS TEZİ

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ABBREVIATIONS

ABS	: Acrylonitrile Butadiene Styrene
ASTM	: American Society for Testing and Materials
ESCR	: Environmental Stress Crack Resistance
HDPE	: High Density Polyethylene
ISO	: International Organization for Standardization
LDPE	: Low Density Polyethylene
LLDPE	: Linear-Low Density Polyethylene
MDPE	: Medium Density Polyethylene
PE	: Polyethylene
PET	: Polyethyleneterephthalate
PMMA	: Polymethylmethacrylate
PP	: Polypropylene
PPO	: Polypropyleneoxide
PTFE	: Polytetrafluoroethylene
PVC	: Polyvinylchloride

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LIST OF SYMBOLS

A	: Cross-sectional area
B	: Specimen width
b	: Slope of S-N curve
b₁	: Width of narrow section of specimen
b₂	: Width of wide section of specimen
E	: Elastic modulus
F	: Force
H	: Thickness of specimen
L	: Distance between test supports
l	: Unit length
l₀	: Initial length
N	: Number of cycle to failure
S	: Stress amplitude
S₀	: Stress intercept value
T_G	: Glass transition temperature
T_M	: Melting temperature
ε	: Strain
σ	: Tensile or compressive stress
σ_f	: Flexural stress
ν	: Poisson's ratio

MECHANICAL PROPERTIES ANALYSIS OF ADBLUE TANK MATERIAL

SUMMARY

The research of the durability of plastics has become a must especially with the increasing use of the plastic components in automotive industry, recent years.

This study is an analysis of the mechanical properties, especially fatigue behavior of plastic material used for AdBlue tanks of Mercedes-Benz EURO IV buses to reduce exhaust emissions.

The first section starts with basic information about the material, followed by the classification of plastics especially emphasizing polyethylene, AdBlue tank material. Furthermore the mechanical properties and the failure types in plastics have been described. While the mechanical properties of plastics are being examined, fatigue behavior and factors affecting fatigue life of plastics have been especially accentuated since they differ from metals. Moreover, rotational molding which is the manufacturing process of AdBlue tanks has been mentioned.

In the following section, standard material tests of linear-low density polyethylene were conducted and the results were evaluated. The tests were done with the standardized specimens under standard testing conditions. In conjunction with the tests, the physical properties of LLDPE, such as tensile strength and elastic modules, were obtained and the differences between the rotational and injection molding, which is the manufacturing process of fatigue test specimens, have been defined.

As mentioned above, fatigue behavior of plastics differ from metals owing to the material properties and external effects. In the last section, it is aimed to identify the fatigue behavior of linear-low density polyethylene to serve as a resource for future analysis. For this purpose, the fatigue tests, executed according to the ASTM D671 test standard, were done and the stress-life (S-N) curve of the material was obtained.

ADBLUE TANK MALZEMESİNİN MEKANİK ÖZELLİKLERİNİN İNCELENMESİ

ÖZET

Son yıllarda araçlarda kullanımı artan plastik komponentler, bu artışa paralel olarak plastiklerde dayanım konusunun incelenmesi gereğini de ortaya koymuştur.

Bu çalışmada ilk olarak, Mercedes-Benz EURO IV otobüslerinde egzoz emisyonlarını azaltmak amacıyla kullanılan AdBlue tanklarının malzemesi olan plastikler hakkında temel bilgilere yer verilmiştir ve plastiklerin sınıflandırılması yapılarak AdBlue tankının malzemesi olan polietilenin özelliklerinden bahsedilmiştir. Bununla beraber plastiklerin mekanik özellikleri ve bu tip malzemelerde görülen kırılma şekilleri anlatılmıştır. Plastiklerin mekanik özellikleri incelenirken metallerden farklılık göstermeleri nedeniyle, yorulma özellikleri ve plastiklerin yorulma davranışına etki eden parametreler üzerinde de durulmuştur. Ayrıca ilk bölümde AdBlue tanklarının üretim prosesi olan döner kalıplama işlemine de değinilmiştir.

Bir sonraki adımda ise plastik tankların malzemesi olan lineer düşük yoğunluklu polietilenin malzeme testleri yapılarak çıkan sonuçlar değerlendirilmiştir. Bu testler standartlara uygun numunelerle ve standart test koşullarında gerçekleştirilmiştir. Bu testler sonucunda malzemenin çekme dayanımı, elastik modülü gibi fiziksel özellikleri elde edilmiş ve bir sonraki adımda ise gerçekleştirilecek olan yorulma testi numunelerinin üretim yöntemi olan enjeksiyonla kalıplama ile döner kalıplama arasındaki farklılıklar ortaya konmuştur.

Plastik malzemelerde yorulma davranışı daha öncede bahsedildiği üzere, gerek malzemenin özellikleri gerekse dış etkenler göz önünde bulundurulduğunda metallerden farklılık gösterir. Bu çalışmanın son bölümünde ise daha ileriki adımlarda gerçekleştirilecek olan yorulma analizlerine kaynak oluşturacak olan, lineer düşük yoğunluklu polietilenin yorulma testlerinin gerçekleştirilmesi amaçlanmıştır. Bu amaçla ASTM D671 standardına uygun yapılan yorulma testleri sonucu söz konusu malzemeye ait gerilme-tekrar sayısı (S-N) ilişkisi bulunmuştur.

1. INTRODUCTION

Plastic's acceptance by the automotive industry can be traced back to the early part of the century, although it was not until advanced, high-performance polymers were developed that plastics became a material of choice for automakers. Since the mid-1980s, automakers have been displacing coated-steel components with plastic ones, especially tanks or engine components. Thus, the use of plastics in automotive applications has risen day after day.

In parallel with this, plastic materials are often used for tanks today because of their lightness, ease for forming complex shapes and corrosion resistance. Today, plastic tanks are used for storage of fuel and AdBlue in Mercedes-Benz EURO IV and EURO V buses.

AdBlue is a solution consisting of high purity urea (typically crystalline) dissolved and suspended within de-ionised water. AdBlue is carried in a separate tank to the fuel and never mix with the fuel. It is injected into the exhaust gases as a post combustion process and it reduces harmful NO_x (Nitrous Oxide) by converting it into Nitrogen and Oxygen.

Polyethylene is used as AdBlue tank material for its corrosion resistance. AdBlue tanks are subjected to variable loads during their entire lives. For this reason, mechanical properties of tank material are often the most important properties and determining these properties provides better understanding about the tank material and its fatigue endurance.

There are many existing, standardized procedures for testing plastics in the form of small specimens. These tests are essential for development and quality control and particularly well-defined in ISO or ASTM standards. Also, by executing the tests of components, it will be determined that the effects of shape and manufacturing methods on the material properties are important for the following applications.

In this study, it is aimed to describe the general mechanical properties of polyethylene especially tensile, flexural and fatigue properties. In the second chapter,

definition of plastics, types of plastics and basic properties of plastics are given in details. Also, the manufacturing process of the AdBlue tanks is mentioned in this chapter. In the third chapter, tensile, flexural and fatigue tests of linear-low density polyethylene and the results of the tests are expressed.

2. THEORY AND BACKGROUND

In this section, definition of plastics, types of plastics and basic properties of plastics are given in details.

2.1 Definition and Structure of Plastics

Plastics, also called polymers, capable of being formed into complex shapes and processing such as traditional materials. A polymer is a material composed of molecules made up of many repeats of monomer.

Engineering plastics are artificially made from carbon-base materials known as polymers. Although polymers are different in many ways from materials such as metals and ceramics, however the fundamentals of polymer science are no more different than the fundamentals of more traditional materials such as metals.

There are various classifications for polymers. While working on polymers, the classifications are generally related to either properties or end-use. The two basic groups of plastic materials are the thermosets and the thermoplastics [1].

2.1.1 Thermosets

Thermosets, or thermosetting plastics, are cured or hardened into a permanent shape. Curing is an irreversible chemical reaction known as cross-linking, which usually occurs under heat.

The cross-linking that occurs in the curing reaction is brought about by the linking of the atoms between or across two linear polymers, resulting in a three-dimensional rigidized chemical structure [1]. An example of this cross-linked structure is shown in Figure 2.1. These cross bonds prevent the chains from slipping and provide a strong and durable structure.

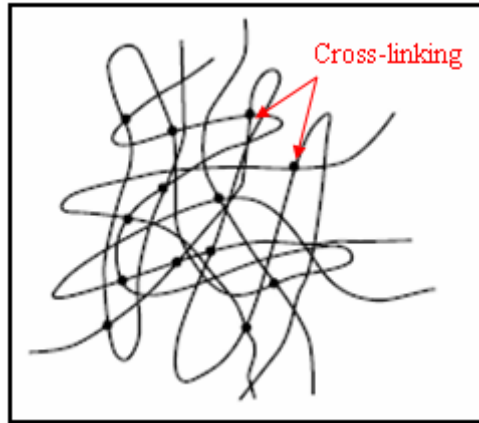


Figure 2.1. Cross-linked Thermoset Molecules

Although the cross-linked part can be softened by heat, it cannot be remelted or restored to the flowable state that existed before cross-linking [1].

Thermoset plastics generally provide some or more of the following advantages:

- High thermal stability
- Resistance to creep and deformation under load
- High rigidity and hardness.

Some typical thermosets are epoxy, polyurethane, silicon and polyimide [2]

2.1.2 Thermoplastics

Thermoplastics differ from thermosets in that they do not cure under heat as thermosets. Thermoplastics consist of long molecules and molecule chains can be thought as independent, intertwined strings as shown in Figure 2.2. Thus they can repeatedly melt and solidified by heating and cooling. During forming, no chemical change generally takes place [2].

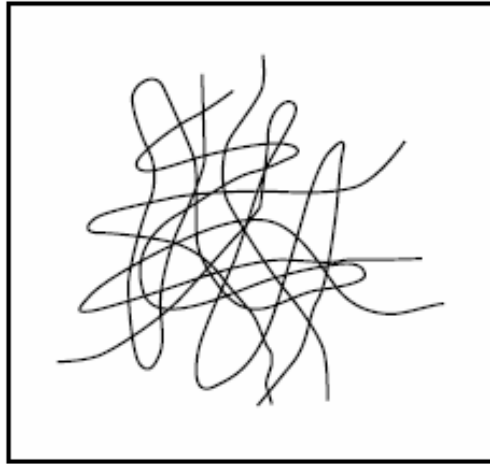


Figure 2.2. Thermoplastics Molecular Chains

The service temperature of thermoplastics is limited by their loss of physical strength. Most of the common thermoplastics are polyethylene, acetal, nylon, acrylic and polyester.

In some thermoplastic, the chemical structure allows the polymer chains to fold on themselves and pack together in an organized manner. This resulting organized regions show the behavior characteristics of crystals. Thermoplastics that have these regions are called crystalline, without this regions are called amorphous. Typical structures of crystalline and amorphous plastics are given on Figure 2.3. All of the crystalline plastics have amorphous region between and connecting the crystalline regions. For this reason, crystalline plastics are often called semi-crystalline [2]. Table 2.1 gives some common examples of semi-crystalline and amorphous thermoplastics.

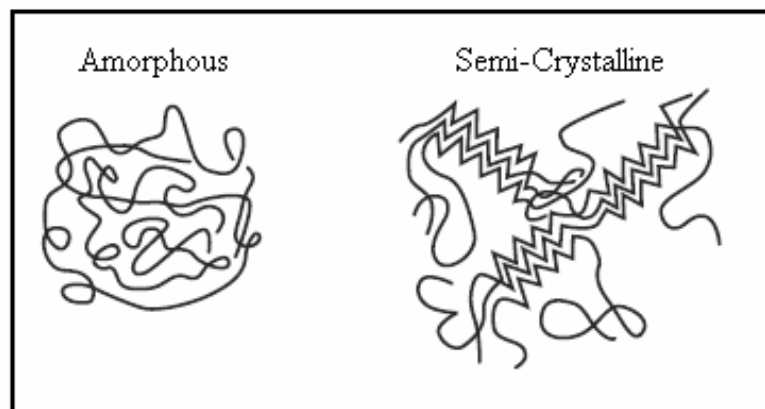


Figure 2.3. Structures of the Amorphous and the Semi-crystalline Thermoplastics

Table 2.1. Typical Amorphous and Semi-crystalline Thermoplastics

Typical Semi-crystalline Thermoplastics	Typical Amorphous Thermoplastics
Polyethylene Acetal Nylon Polypropylene Polyester	Polystyrene ABS Polycarbonate PVC

Amorphous thermoplastics have a structure that shows no regularity and soften continuously as heat applied. They do not flow easily as molten crystalline thermoplastics in the molting process. Semi-crystalline thermoplastics show local regular crystalline structures in their solid state [3].

Polyethylene and polypropylene can be considered as the first two members of a large group of polymers based on the ethylene structure [1]. As mentioned before, AdBlue tanks are made of polyethylene material which is focused on this study.

2.1.2.1 Polyethylene

Polyethylene (PE) is created through polymerization of ethylene. PE is the simplest of all polymers with just two carbons and four hydrogens. PE consists of only carbons and hydrogens, usually with high molecular weights and so it is relatively insensitive to most solvents. This is an advantage when PE is used for applications where inertness of the container is critical.

Polyethylene is one of the best-known plastics and come in three main classifications based on density: low, medium and high [1]. However, in some references, linear-low density polyethylene (LLDPE) is mentioned as another common type of PE. The density ranges are given on Table 2.2.

Linear polyethylene, as the name suggests, has very little branching along the polymer chains. LLDPE polymers, with little-long chain branching, have much greater elongation than low-density polyethylene (LDPE). LLDPE has higher tear, tensile and impact strength so this allows stronger products to be produced with less material [4]. Principally, because of these advantages, LLDPE is chosen as AdBlue tank material which has a density of 0.934 g/cm^3 .

Table 2.2. Classification of Polyethylene

Polyethylene Type	Density (g/cm ³)
Low Density (LDPE)	0.910 - 0.925
Linear-Low Density (LLDPE)	0.918 - 0.940
Medium Density (MDPE)	0.926 - 0.940
High Density (HDPE)	0.941 – 0.965

Density directly affects some material properties in a good or bad way, as shown in Figure 2.4. Polyethylene has good toughness near-zero moisture absorption, excellent chemical resistance, low friction coefficient and ease of processing. Only a few thermoplastics have these excellent properties of polyethylene [1].

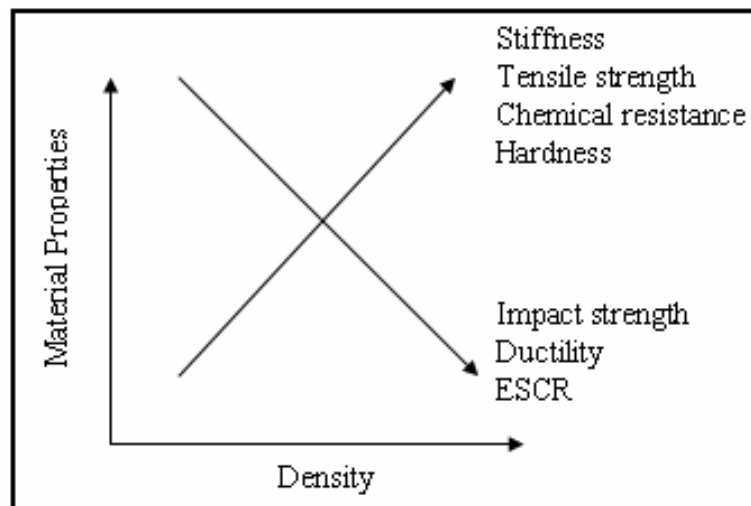


Figure 2.4. Effect of the Density

Traditional markets for polyethylene are wire and cable coating, liquid tanks, pipe and tubing and packaging films.

2.2 Mechanical Properties of Plastics

Any force or load acting on a body results in stress and strain in the body. Stress represents the intensity of the force at any point in the body and is measured as the force acting per unit area of a plane. The deformation in shape or dimensions of the body, resulting from the stress is called strain [5]. Strain is expressed in

dimensionless units, such as $\mu\text{m}/\text{m}$, or in percentage. Tensile strain, ϵ , is expressed as elongation per unit length, l

$$\epsilon = \Delta l / l = (l - l_0) / l \quad (2.1)$$

If the applied force or load, F , is tensile or compressive, the resulting tensile or compressive stress, σ is defined by

$$\sigma = F/A \quad (2.2)$$

where A is the cross-sectional area, perpendicular to the direction of the force.

When the ideal elastic body is subjected to tensile, the modulus of elasticity E , is expressed as

$$\sigma = E \cdot \epsilon \quad (2.3)$$

2.2.1 Short-Term Tensile Behavior of Plastics

The stress–strain behavior of plastics measured at a constant rate of loading provides a basis for quality control and comparative evaluation of various plastics. The diagram shown in Figure 2.5 is the most typical stress-strain curve of the metals and plastics. It can be seen that plastic materials do not have a distinct linear response like metals.

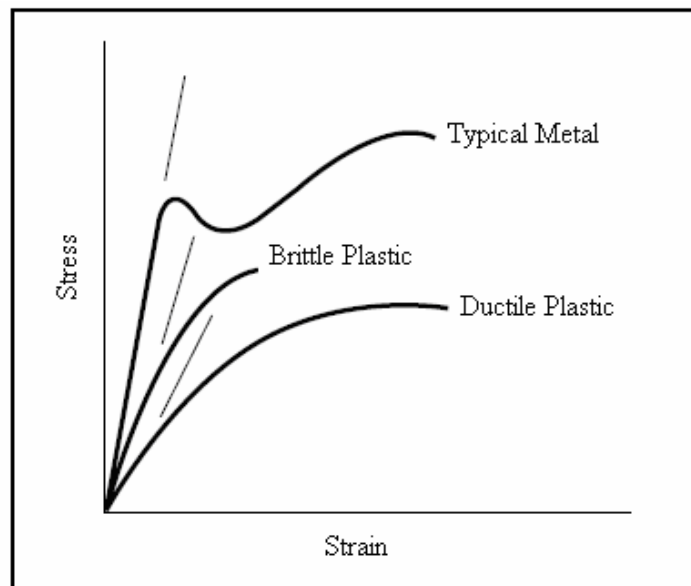


Figure 2.5. Tensile Responses of Metal & Plastic

Most engineering metals exhibit a linear strain response to increasing stress below yield point and the strain is fully recoverable when the metal component is unloaded. However when a plastic is pulled in tension it undergoes elastic strain. The resulting stress-strain curve is nonlinear except, in some cases, at very low strain. Also, when the stress is relaxed, not of all the strain is recovered immediately. However if the plastic remains unloaded for several minutes and hours some additional strain may be recovered, but there may be still be a measurable permanent strain or “set” [1]. This variation in stress-strain behavior is represented in Figure 2.6.

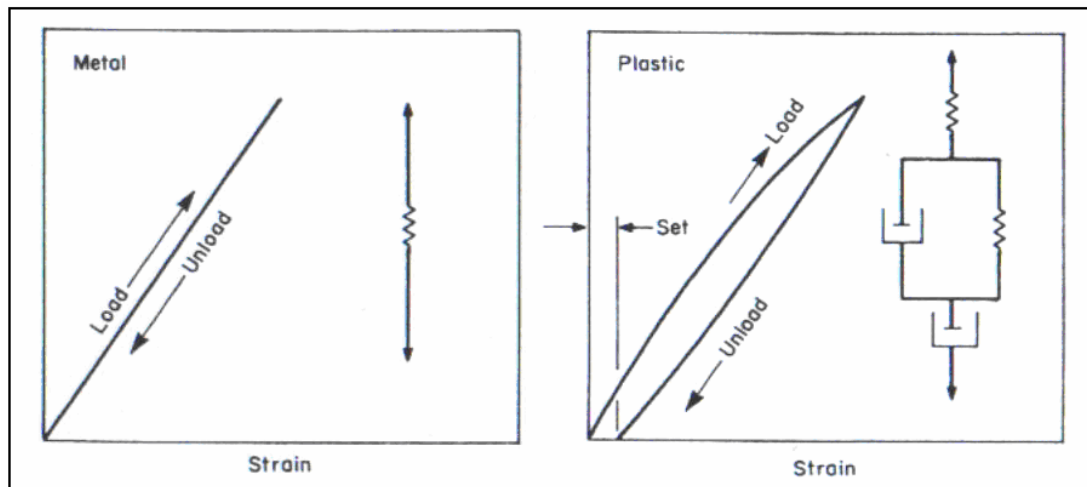


Figure 2.6. Strain-Stress Characteristics of Metals and Plastics

In general an increase in temperature of about 10°C will not significantly change the performance of the metal, but it will probably result in considerably higher strain for the plastic. Likewise, a change in loading rate for the metals will not alter the stress-strain characteristics. Typical effects of temperature and loading rate are shown in Figure 2.7 for a plastic loaded in tension [1].

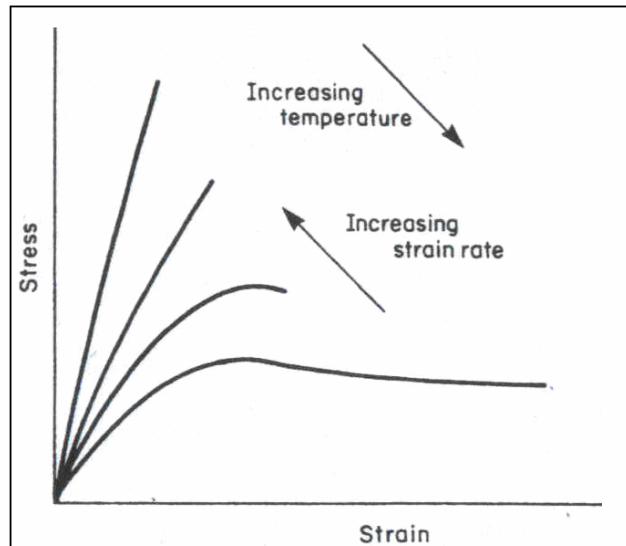


Figure 2.7. Influence of Temperature and Loading Rate on Plastics

In plastics failure mechanism changes according to many effects for instance internal structure, temperature and loading rate etc. There are two general types of fracture in: brittle and ductile.

2.2.1.1 Brittle Failure

In brittle materials, failure typically occurs at very low strains (perhaps 1% or less) and it is generally associated with amorphous thermoplastics below their glass transition temperature, T_G [6] that is a temperature range at which a polymer undergoes a reversible change from a viscous or rubbery behavior to a brittle or glassy behavior. Figure 2.8 shows the stress-strain curve of a typical brittle plastic.

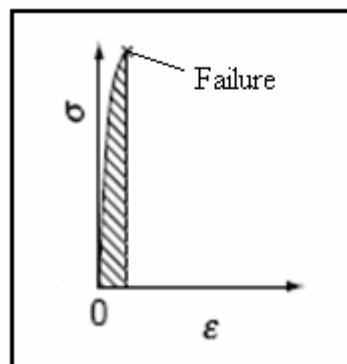


Figure 2.8. Stress-Strain Curve of a Brittle Plastic

The area under the stress-strain diagram is measure of the energy a sample can absorb before it breaks. The energy to cause such failures is small and is stored in the material as elastic strain energy [5].

2.2.1.2 Ductile Failure

A ductile failure takes place with semi-crystalline thermoplastics at temperatures between the glass transition temperature, T_G , and the melting temperature, T_M [6]. Large permanent deformation has occurred before failure. As a result of the permanent deformation, the two fracture surfaces do not match, and the cross-sectional area at the location of fracture is reduced from the original value. A ductile failure is a succession of several events, as clearly shown in Figure 2.9.

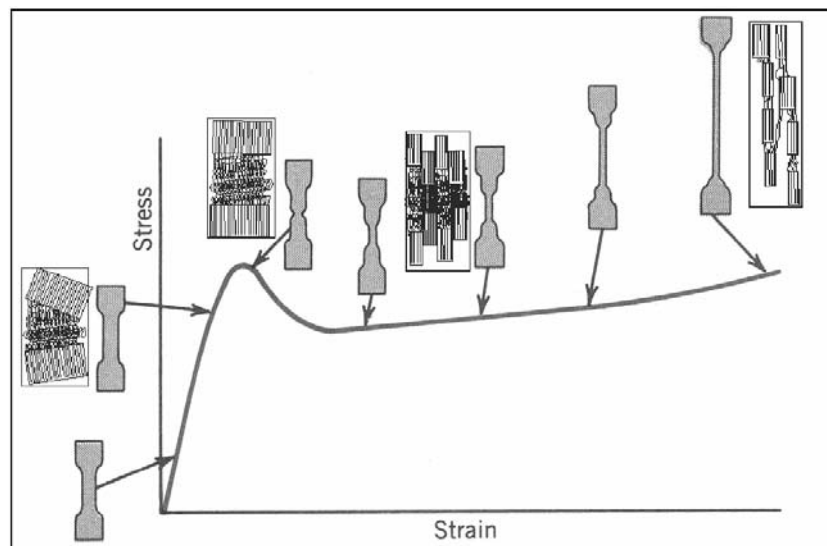


Figure 2.9. Stress-Strain Curve of a Ductile Plastic

Necking on ductile plastics, is clearly seen in Figure 2.9. First the amorphous ties between crystalline regions completely extend. As deformation continues, crystalline regions get aligned in the direction of draw and slide over each other. After forming of an alternating crystal blocks, necking starts close to the yield point.

2.2.2 Fatigue Behavior of Plastics

Fatigue is the phenomenon of the materials failing under cyclic loads below their static strength, has been extensively investigated with respect to metals but not plastics. Some work has been done recently on thermosetting plastics and some data have been published for a few thermoplastics. However, virtually no investigation

has been undertaken to understand the fatigue behavior of thermoplastics, especially polyethylene.

Fatigue testing results are plotted as stress amplitude, S , versus number of cycles to failure, N . These graphs are usually called S-N curves (also known as the Wöhler curve). A schematic view of S-N curve comparing with plastics and metal is shown in Figure 2.10.

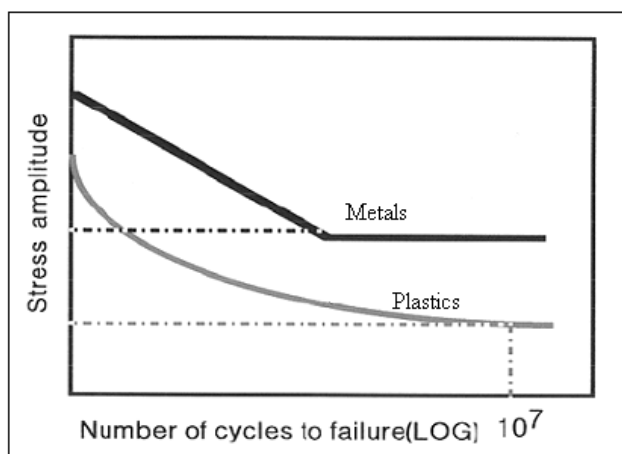


Figure 2.10. S-N Curves of Metals and Plastics

A term, “Fatigue Endurance Limit”, have been defined to represent the maximum cyclical stress that a material can be subjected to and still have infinite life. The endurance limit can be related to the tensile strength. The response of plastics to cyclical stress is more complex than the response of metals, and plastics do not have a distinct endurance limit like metals. The fatigue endurance limit is accepted at 10^7 cycles for the plastic materials.

Figure 2.11 presents S-N curves for several thermoplastic and thermoset polymers which are tested according to ASTM D-671 test method. These tests are performed at a 30 Hz frequency and about a zero mean stress, σ_m [6]. As seen in Figure 2.11, PE has lower fatigue strength than other plastics.

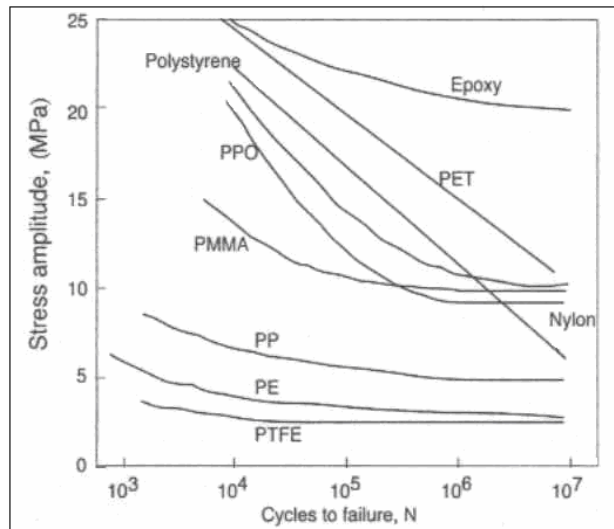


Figure 2.11. Stress-Life Curves for Several Thermoplastics and Thermosets

2.2.2.1 Factors Affecting Fatigue Life

Fatigue in plastics is strongly dependent on environment, the temperature, the frequency of loading, the surface etc. It is interesting to point out in Figure 2.11 that thermoset polymers show higher fatigue strength than thermoplastics. An obvious cause for this is their greater rigidity. However more important is the lower internal damping or friction, which reduces temperature rise during testing.

Temperature rise during testing is one of the main factors that lead to failure when experimentally testing thermoplastic polymers under cyclic loads. The heat generation during testing is caused by the combination of internal frictional or hysteretic heating and low thermal conductivity. At low frequencies, the heat can be removed from the specimen by conduction.

On the other hand, if the frequency or stress level is increased even further, the temperature will rise to the point at which the test specimen softens and ruptures before reaching thermal equilibrium. For this reason, increasing the frequency causes a decrease in fatigue life. Frequency and stress level effect can be seen in Figure 2.12. [7]. These S-N curves belong to polytetrafluoroethylene which is a synthetic thermoplastic and performs significantly better than nylon and acetal.

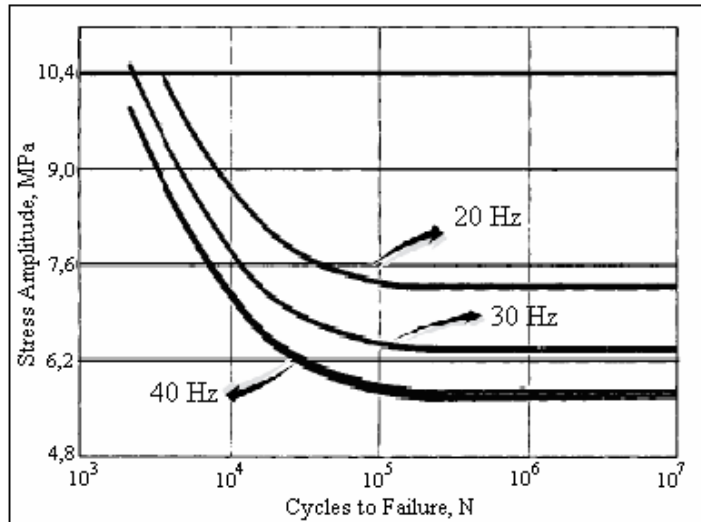


Figure 2.12. Effect of Frequency on Fatigue Life

Another main parameter which affects fatigue life on plastics is environment, especially the environment temperature. In general an increasing in temperature will not significantly change the fatigue performance of the metals, but it will probably result in considerably lower fatigue strength for the plastics. As it is shown in Figure 2.13, the fatigue life of plastics is related to the environmental temperature and increasing the temperature leads up to a poor fatigue life.

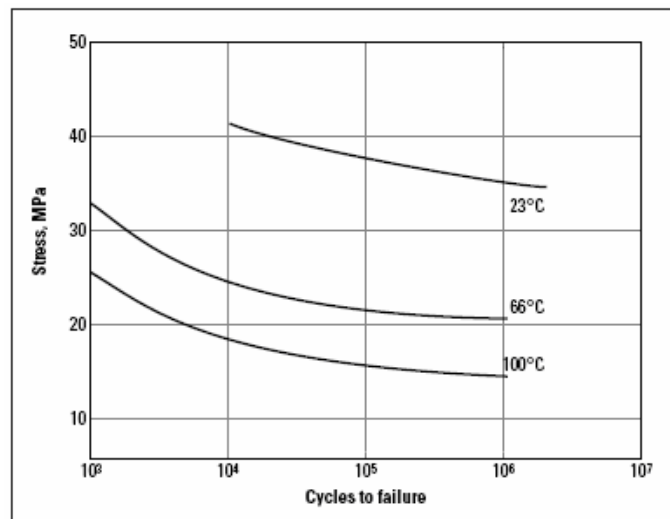


Figure 2.13. Effect of Temperature on Fatigue Life

In Figure 2.13 the S-N curves obtained from testing the acetal specimens which is a kind of thermoplastic, having a rigid behavior than polyethylene [8].

As mentioned before stress level have a great influence on the fatigue life of the component. The biggest effect on increasing stress level is stress concentrated regions on the component. For example a circular hole or a distinct corner on the part acts as a stress concentrator.

Most of the components that are subjected to cyclic loading have other loads or stresses applied to them, leading to non-zero mean stress values. Also these loads can be cause creep.

Also the production process of the component is very important for fatigue life. Because of the surface irregularities and scratches, crack initiation at the surface is more likely in machined components than in molded parts.

2.3 Manufacturing Plastic Parts

There are a variety of processing alternatives for plastics. Thermosets and thermoplastics offer the options of extrusion, injection, blow and rotational molding. The manufacturing process to be used for making a plastic component should be selected when the design and material are being determined. Design, material and process should be considered simultaneously because they are so closely interrelated. Before selecting the manufacturing process, the entire process should be considered.

The different processes are capable of producing parts with different physical properties. For plastic AdBlue tanks, rotational molding is the main processing technique because of the applicability to the most thermoplastics, especially polyethylene.

2.3.1 Rotational Molding

Rotational molding, known also as rotomolding, is a process for manufacturing hollow plastic products like containers or tanks. The product is formed from liquid or powdered thermoplastic resins inside a closed mold while the mold rotating biaxially.

Rotational molding is best suited for large and hollow products requiring stress-free strength, complicated curves, uniform wall thickness, and a good finishing. Rotational molding offers a number of advantages; [4]

- Stress free and seamless parts
- Large hollow shapes of relatively simple shape
- Low tooling and machinery cost
- Quick mold changes
- Integrate metal inserts easily.

There are five basic steps in rotational molding: preparing, loading, molding or heating, cooling and unloading as shown in Figure 2.14.

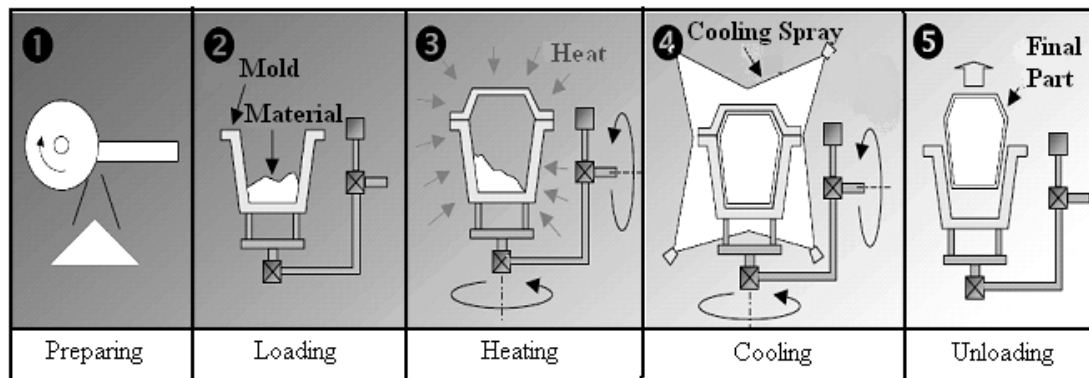


Figure 2.14. Conventional Rotational Molding Process

Material is put into the mold as powder or liquid form. If the material is in granule form it must be grinded before the loading. In loading stage, the powdered plastic is charged into a hollow mold. The mold halves then are clamped shut and moved into the oven.

In the oven, the heat penetrates the mold, causing the plastic become tacky and stick. On most units, the heating is done by air or by a liquid of high specific heat. Because of the mold continue to rotate while heating, the plastic is gradually distributed on the mold walls.

When the part has been formed, the molds move to a cooling chamber. Cooling process is generally done with cold water spray or forced cold air. While cooling, the mold continues to rotate to ensure that the part does not sag away from the mold.

Finally the mold is opened and the part is removed. Process time can change from 5 to 30 minutes according to the part size. But typically the cycle time range from 7 to 15 minutes [4].

3. MATERIAL TESTS

The material selection is quite based on mechanical properties such as tensile and flexural properties or fatigue resistance. The mechanical properties are the one of important properties for plastics because the service life of a component involves mostly mechanical loading. It is identical for plastic tanks. They are also mainly subjected to mechanical loads in their entire lives. For a thorough understanding of mechanical properties, tests and the effects of adverse conditions are extremely important. Therefore, some tests were conducted for determining the mechanical properties of linear-low density polyethylene which is the material of AdBlue tanks used on buses.

3.1 Determination of Tensile Properties of Plastics (ISO 527-1)

The ISO 527-1 standard test method specifies the general principles for determining the tensile properties of plastics and plastic composites under defined conditions. A tensile stress-strain curve of a plastic can be plotted with the help of this test method.

The tensile behavior of plastics measured at a constant rate of loading provides a basis for quality control and comparative evaluation of various plastics. The diagram shown in Figure 3.1 is most typical of that obtained in tension for a constant rate of loading for different densities of polyethylene.

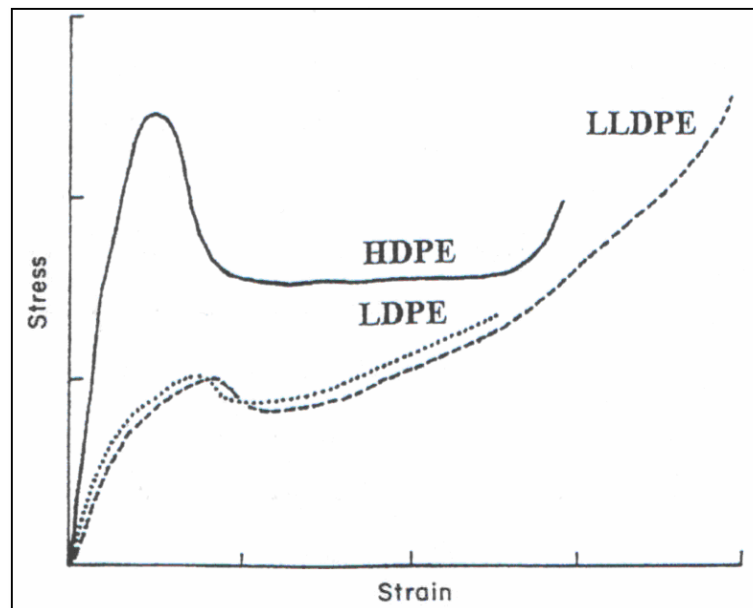


Figure 3.1. Tensile Response of Various Polyethylene Types

In the diagram, stress is plotted against strain. Even for different plastics the nature of the curves will be similar, but they will differ in the numerical values. Ultimate strength, elongation, and elastic modulus can be obtained from the stress-strain curve [9].

3.1.1 Test Apparatus

The tensile testing machine, shown in Figure 3.2, has a fixed and a movable head. A velocity controlled drive mechanism is used. An extensometer is used to determine the elongation between two specific points located on the specimen, as the specimen is stretched.

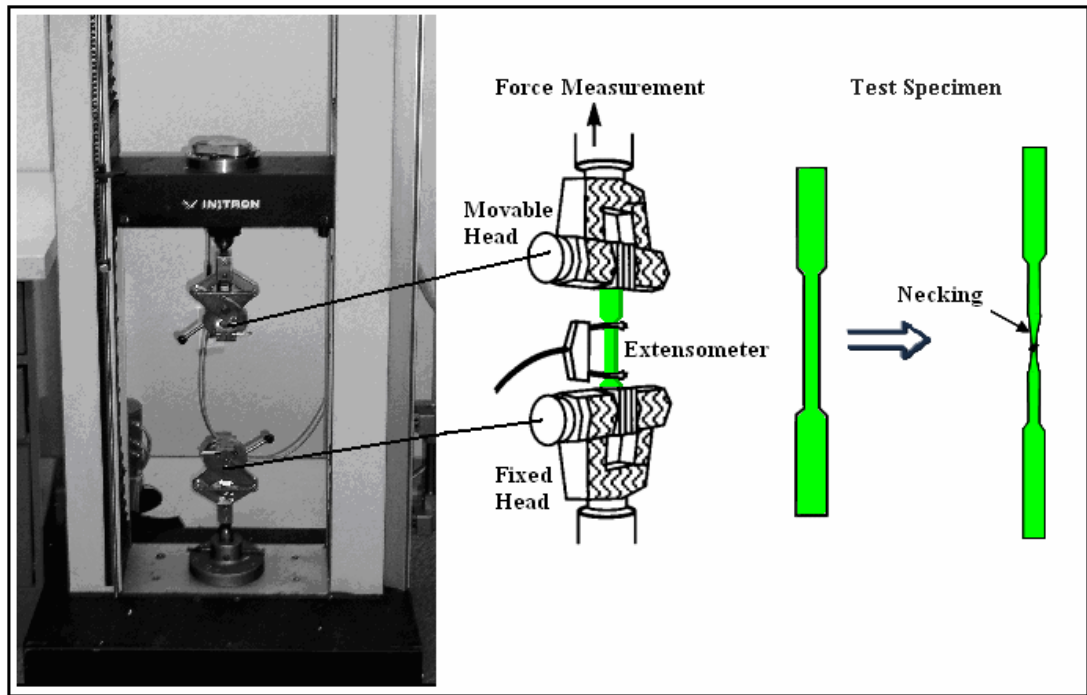


Figure 3.2. Tensile Testing Machine

3.1.2 Test Specimen and Conditioning

In tensile tests, test specimens can be prepared many different ways. Most often, they are injection molded. Test specimen dimensions vary depending upon the requirements as described in standard. Since the tensile properties of some plastics change rapidly with changes in temperature, the tests should be conducted in standard laboratory conditions of $23 \pm 2^\circ\text{C}$ and 50 % humidity [10].

In tensile test of polyethylene, the specimens were produced with injection molding and the test was conducted with Type 1B specimens, as mentioned in ISO 527 standard. The test specimen shape and dimensions are shown in Figure 3.3 and Table 3.1 [10].

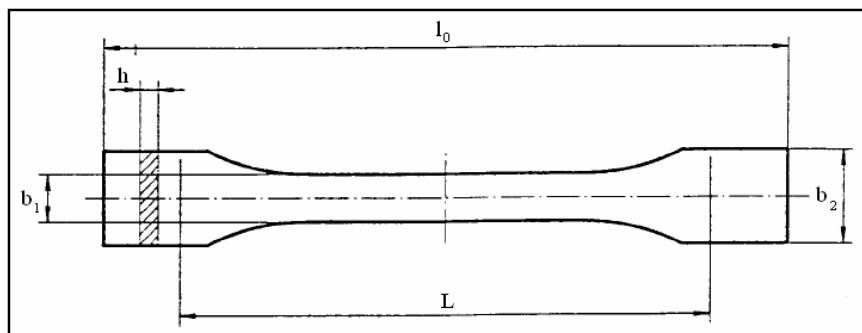


Figure 3.3. Tensile Test Specimen (Type 1B)

Table 3.1. Dimensions of Tensile Test Specimen (mm)

l_0	Specimen Length	140
L	Distance Between Head	115
b_1	Width of Narrow Section	10
b_2	Width of Wide Section	20
H	Thickness of the Specimen	4

3.1.3 Test Procedure

The test specimen is positioned vertically in the grips of the testing machine. The grips are tightened firmly to prevent slippage. There are basically nine different testing speed specified in the ISO 527 standard. As the specimen elongates, the resistance of the specimen increases and the load value is recorded. The tensile strength at yield and at break is calculated with equation (2.2). Also, the tensile modulus as mentioned in Section 2.2 is calculated with the following equation.

$$E = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \quad (3.1)$$

In equation (3.1), σ_2 and σ_1 are the measured stresses when the strains values become $\varepsilon_2=0,0025$ and $\varepsilon_1=0,0005$ [10].

To specify the tensile properties of polyethylene, this test was conducted at a constant test speed, 1 mm/min. Five injection molded specimens were tested and the stress-strain curve of each specimen was obtained as shown in Figure 3.4.

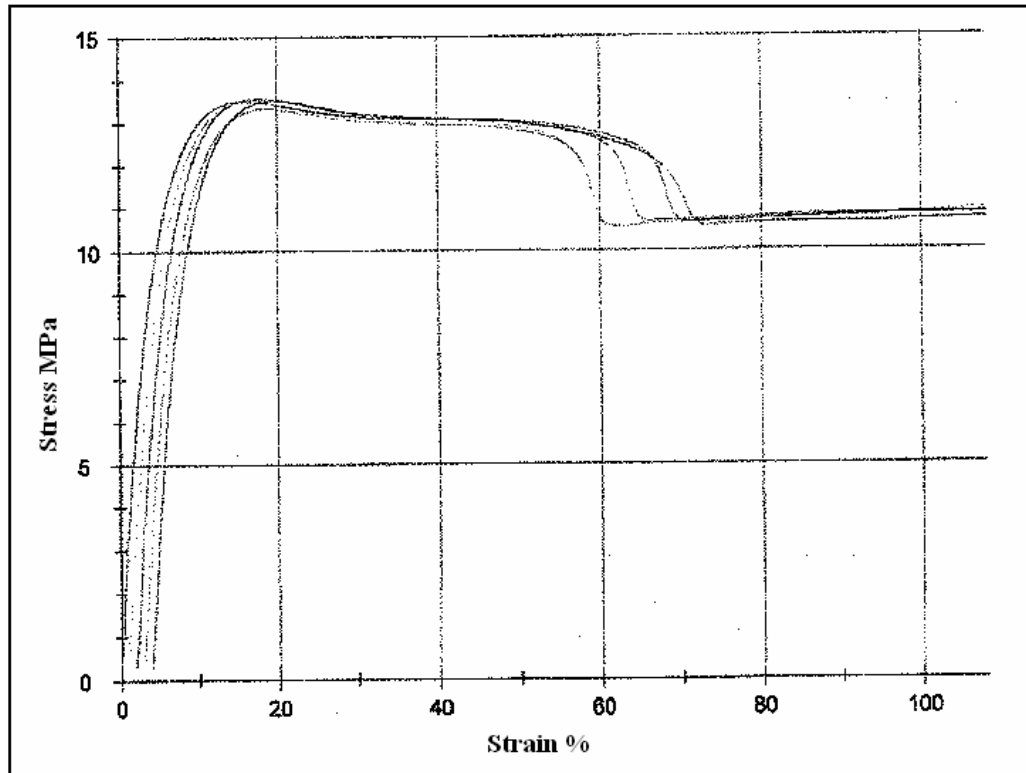


Figure 3.4. Stress-Strain Curve of Polyethylene Tensile Test Specimens

As seen in Figure 3.4, the elongation of linear-low density polyethylene is extremely high and all of the specimens did not break within the limits of the tensile testing machine. It is seen in Table 3.2 that, there is a difference between the tensile modulus. The manufacturer value is higher than the test values. Manufacturing process of the specimens, test speed, temperature and humidity can be the reasons of this difference. But at yield point, the tensile stress values are almost same. Therefore, the test values such as tensile strength obtained from these tensile tests which were conducted in laboratory under standard test conditions will be used before starting fatigue test.

Table 3.2. Tensile Properties of Linear-Low Density Polyethylene

Test Numbers	Tensile Modulus (MPa)	Tensile Stress at Yield (MPa)
1	128.2	13.52
2	344	13.49
3	231.28	13.58
4	390.02	13.34
5	216.53	13.52
Average Value	261.97	13.49
Manufacturer Value	650	18

3.2 Determination of Flexural Properties of Plastics (ISO 178)

Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. Flexural properties are reported and calculated in terms of the maximum stress and strain that occur at the outside surface of the test specimen. Many polymers do not break under flexure even after a large deflection that makes determination of the ultimate flexural strength impractical for many polymers. In such cases, the common practice is to report flexural yield strength when the maximum strain in the outer surface has reached at a specific value [9].

There are many advantages of flexural tests over tensile tests. If a component is subjected to bending in its entire service life, it is more relevant to make a flexural test over this material. Likewise, AdBlue tank walls are generally subjected to bending because tanks are perpendicular to the vehicle movement direction as seen in Figure 3.5. Thus, it can be beneficial to determine the flexural behavior of linear-low density polyethylene. Another advantage of the flexural tests is that at small strains the actual deformations are large to be measured accurately.

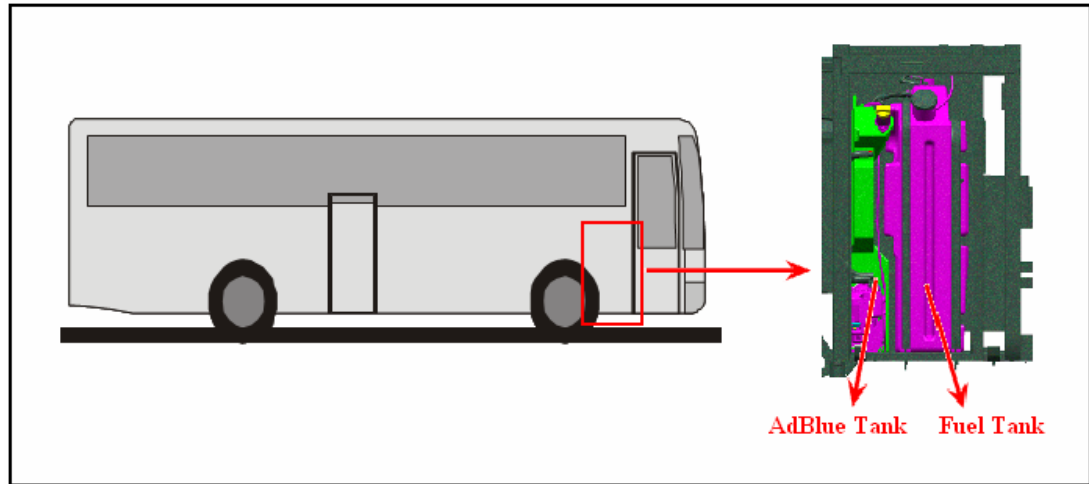


Figure 3.5. The Location of AdBlue Tank in Bus

3.2.1 Test Apparatus

There are two basic methods that cover the determination of flexural properties of plastics. Method 1 is a three point loading system which is utilizing center loading on a simple supported beam. A bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midpoint of the specimen. This method is especially useful in determining flexural properties for quality control and specification purposes.

Method 2 is a four point loading system which is utilizing two load points equally spaced from the supports with a distance between load points of one third of the support span.

While determining the flexural properties of linear-low density polyethylene, three point bending test method (method 1) was used as shown in Figure 3.6. The loading nose and support must have cylindrical surfaces. The radius of the nose and supports should be 5 mm to avoid stress concentration as mentioned in test standard [11]. In flexural test of the linear-low density polyethylene, measurement of deflection was done with the help of the strain gauges which were applied on the outer surface of the specimens.

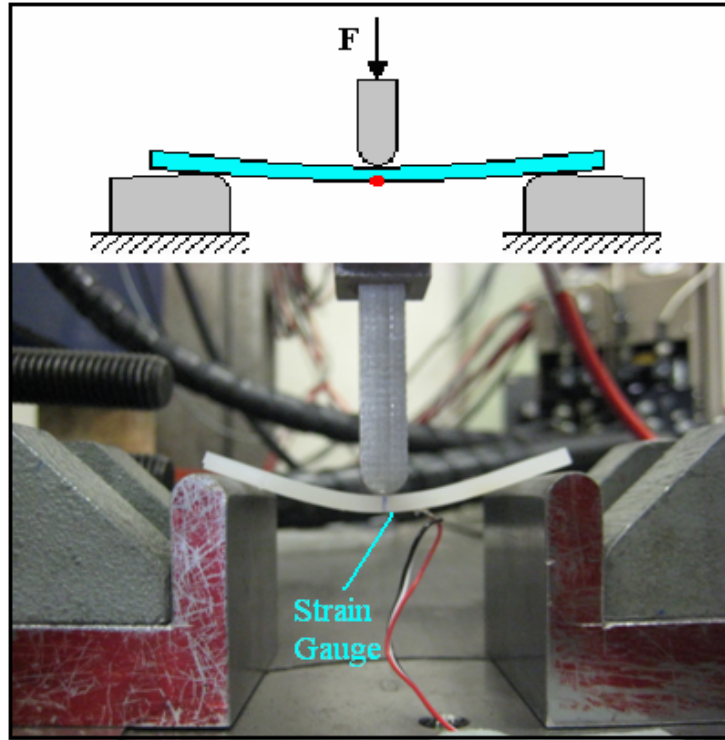


Figure 3.6. View of the Specimen Arrangement for Flexural Testing

3.2.2 Test Specimen and Conditioning

The specimens used for flexural testing are bars of rectangular cross section and are cut of from sheets, plates or molded shapes. In flexural tests of linear-low density polyethylene injection and rotational molded specimens were used. Test specimen dimensions were same in both manufacturing process as shown in Figure 3.7 and Table 3.3. Tests were conducted in standard laboratory conditions of $23 \pm 2^\circ\text{C}$ and 50 % humidity [11].

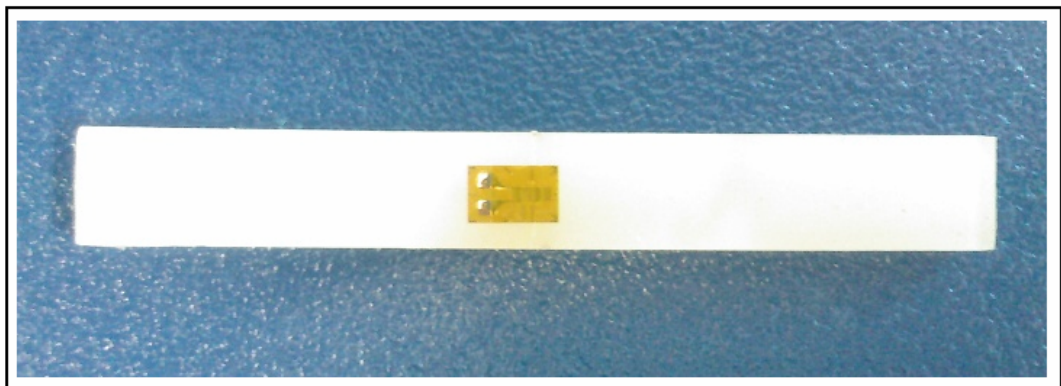


Figure 3.7. Flexural Test Specimen

Table 3.3. Flexural Test Specimen Dimensions

l_0	Specimen Length	80
L	Distance Between Supports	64
B	Specimen Width	10
H	Thickness of the Specimen	4

3.2.3 Test Procedure and Calculations

The test is initiated by applying the load to the specimen at a constant speed. There are nine different test speed specified in ISO 178 standard. As the load is applied, the deflection of the specimen increases and the load and the strain value are recorded. Many polymers do not break under flexure. In such cases the test is stopped when the maximum strain in the outer surface of the specimen has reached 5%.

The maximum stress is related to the load and sample dimensions and is calculated using the following equation; [11]

$$\sigma_f = \frac{3.F.L}{2.B.H^2} \quad (3.1)$$

After calculating the maximum stress a stress-strain curve can be plotted.

The flexural test of linear-low density polyethylene was conducted at a constant speed of 2 mm/min. Besides the injection molded specimens, also rotational molded specimens were tested to compare both manufacturing process. Throughout the tests five injection and five rotational molded specimens were tested. The strain value at the outer surface of the specimen was measured by using strain gauges and the stress-strain curves were plotted for injection and rotational molded specimens as shown in Figure 3.8. While collecting strain data on specimens, 350 Ohms, type FAE-A6163P-35-S6EL, BLH strain gauges were used, as shown in Figure 3.7.

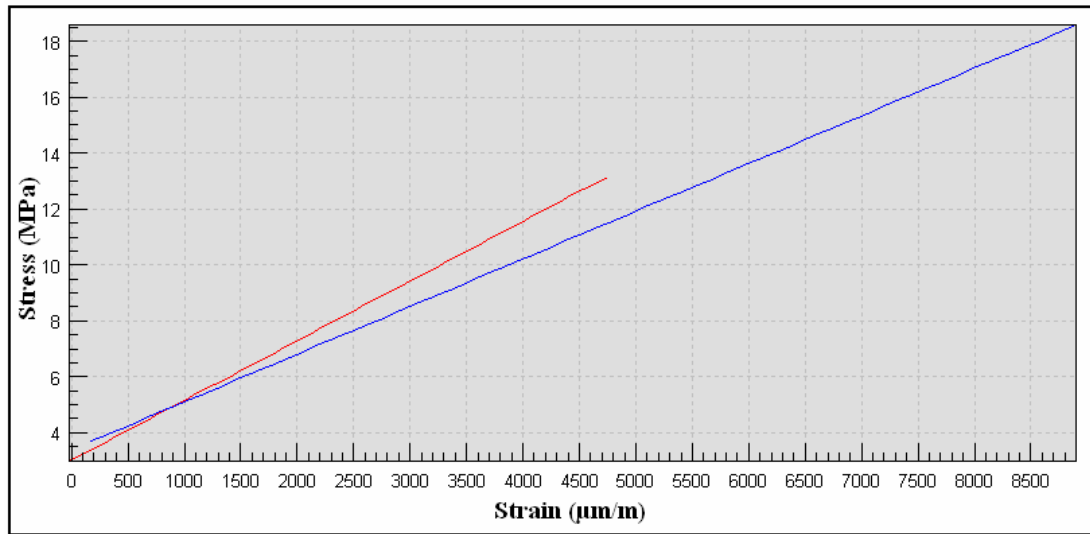


Figure 3.8. Flexural Stress-Strain Plot

As seen in Figure 3.8, only elastic regions of the stress-strain curves were analyzed because, on AdBlue tank tests at Hydropuls, measured strain range on critical locations is approximately 2000-3000 µm/m. Thus, it is enough to analyze the elastic regions of the curves below 5000 µm/m. Both curves have small difference in main slopes. The slopes are 0,00213 for injection and 0,00170 for rotational molded specimens. Therefore they behave almost same in bending and injection molding can be used for manufacturing the complex shaped specimens like in flexural fatigue test.

3.3 Standard Test Method for Flexural Fatigue of Plastics by Constant Amplitude of Force (ASTM D671)

Flexural fatigue test covers the determination of the effect of repetitions of the same magnitude of flexural stress on plastics by fixed-cantilever type testing machines, which produce a constant-amplitude-of force on the test specimen each cycle. By using this test method it can be understood that how the specimen will perform under similar conditions in actual use.

The results are suitable for direct application in design only when all design factors including magnitude of stress, size and shape of part, environment and part temperature, heat transfer conditions, cyclic frequency, and environmental conditions are comparable to the test conditions.

In literature, there is not any specific S-N curve which belongs to the material of the AdBlue tanks, linear-low density polyethylene. As mentioned before, AdBlue tanks are generally subjected to flexural stresses because tanks are perpendicular to the vehicle movement direction. Therefore, it is beneficial to obtain an S-N curve of the linear-low density polyethylene for the durability analysis of the tanks.

3.3.1 Test Apparatus

A fatigue testing machine of the fixed-cantilever, repeated-constant-force type, is shown in Figure 3.9. In linear-low density polyethylene fatigue tests, MTS 858 Mini Bionix servohydraulic test unit was used.



Figure 3.9. Flexural Fatigue Test Setup

In this machine, the specimen should be held at its big-end as a cantilever beam and bent by a constant load applied through a connection, fastened to the small-end [12] (Figure 3.10).

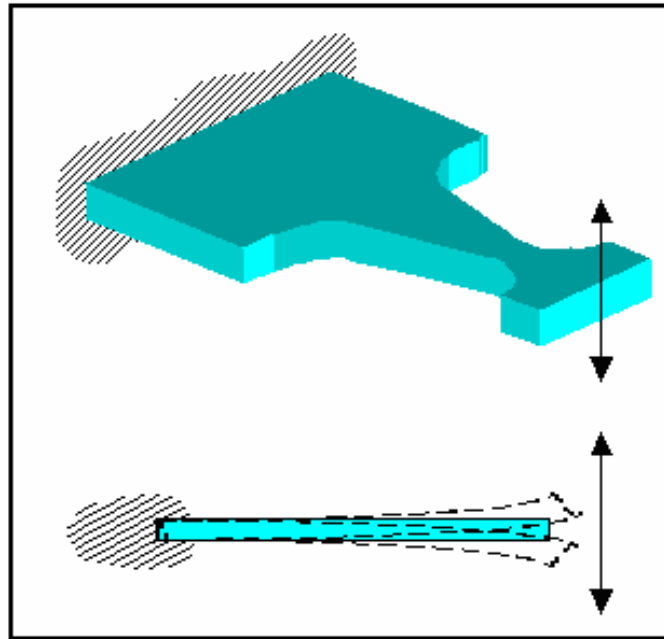


Figure 3.10. Schematic View of Execution of Fatigue Test

3.3.2 Test Specimen

Selection of a particular specimen will depend upon specimen thickness and the stress range over which the measurements are to be made. The triangular form of the specimen provides uniform stress distribution [12]. The specimen type which was used in plastic fatigue tests is shown in Figure 3.11. Specimens were manufactured with injection molding according to the dimensions specified in Figure 3.11. The reason of choosing injection molded specimens is that, it is more relevant to produce large number of specimens with injection molding and also manufacturing this kind of specimen with rotational molding is difficult because of the small shape of the specimen. The thickness of the specimen was selected as 7mm which reflects the real wall thickness of the plastic AdBlue tanks used in buses. The strain gauges were applied to the specimens to measure the strain variation throughout the fatigue tests. Strain gauges have the same specifications as used in flexural tests.

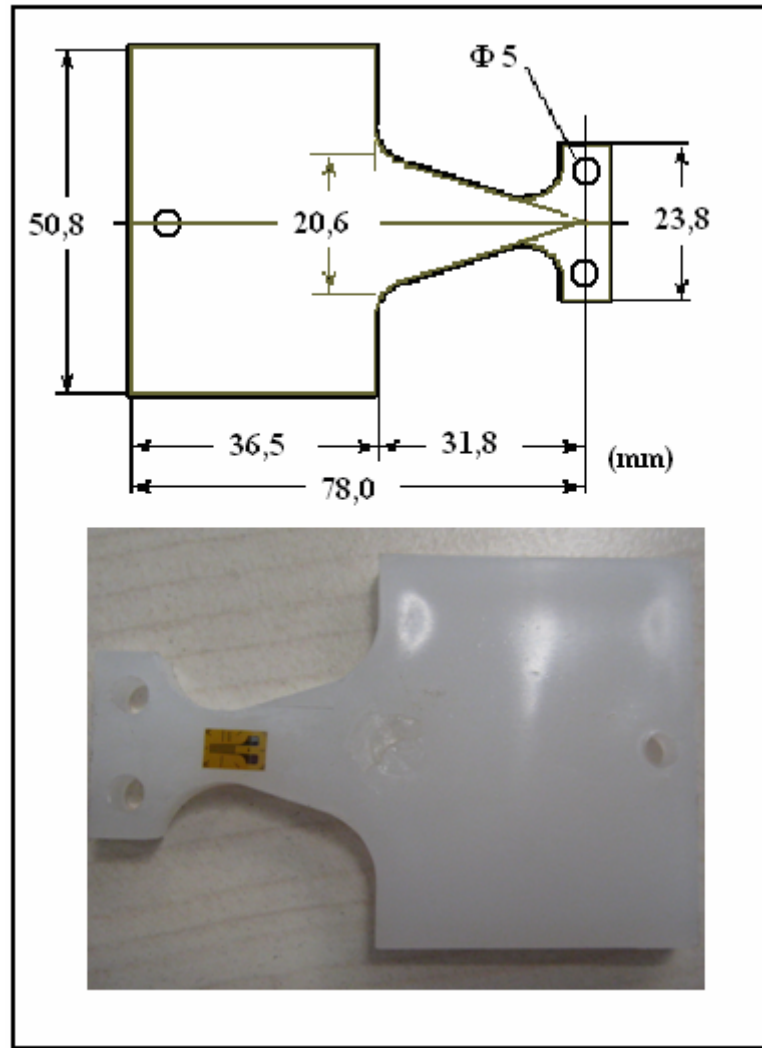


Figure 3.11. Flexural Fatigue Test Specimen

3.3.3 Test Procedure and Calculations

To determine the effect of various load conditions at different positions, a finite element analysis was executed over a solid model of the test specimen before the fatigue test, as seen in Figure 3.12.

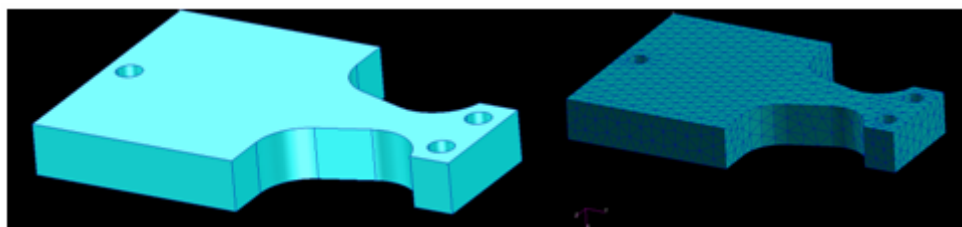


Figure 3.12. Solid and Meshed Model of the Test Specimen

Before the finite element analysis, material parameters (elastic modulus, Poisson's ratio) should be set correctly in the software. For this purpose, the following material properties were taken from the material supplier. MSC Patran was used for this finite element analysis as software.

$$E = 700 \text{ MPa}$$

$$\nu = 0,33$$

The boundary conditions were defined as the specimen position in the fatigue tests as shown in Figure 3.13.

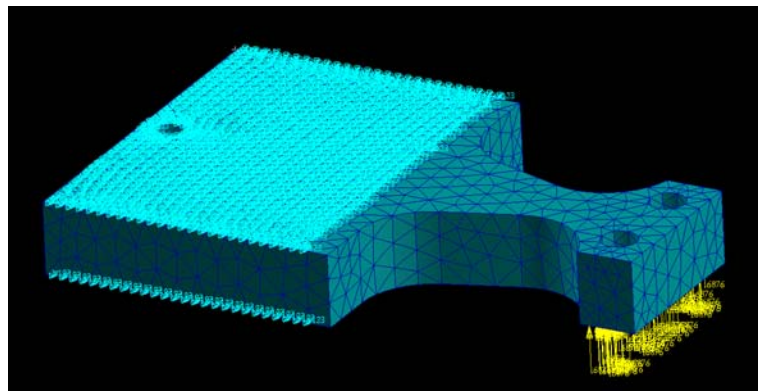


Figure 3.13. Boundary Conditions

According to the finite element analysis, for the various loading conditions the analysis result are shown in Figure 3.14. As the results show that the maximum stresses are seen on the neck region of the specimen [13]. In fatigue tests it is anticipated that the failure occurs on this area.

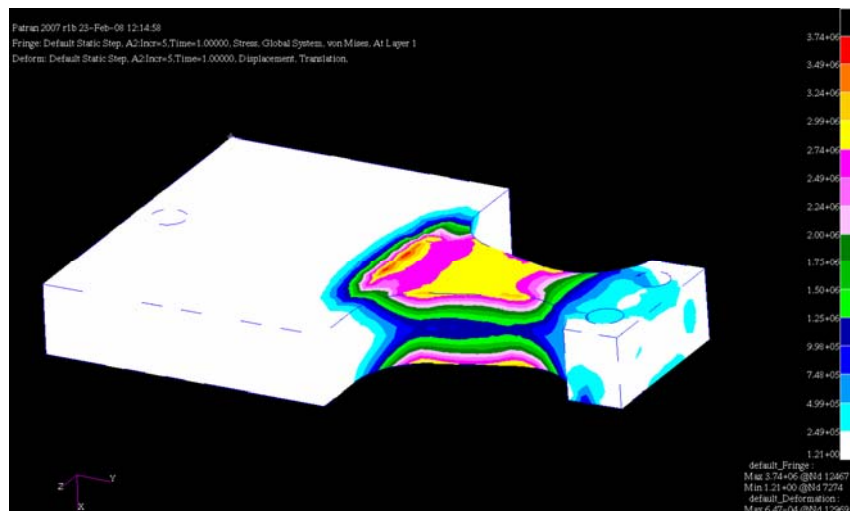


Figure 3.14 Von Misses Stress at 30 N

After applying the forces vary from 30 to 60 N, the stress values on the neck region of the specimen are given in Figure 3.15 and Table 3.4. The other values calculated by the software [13].

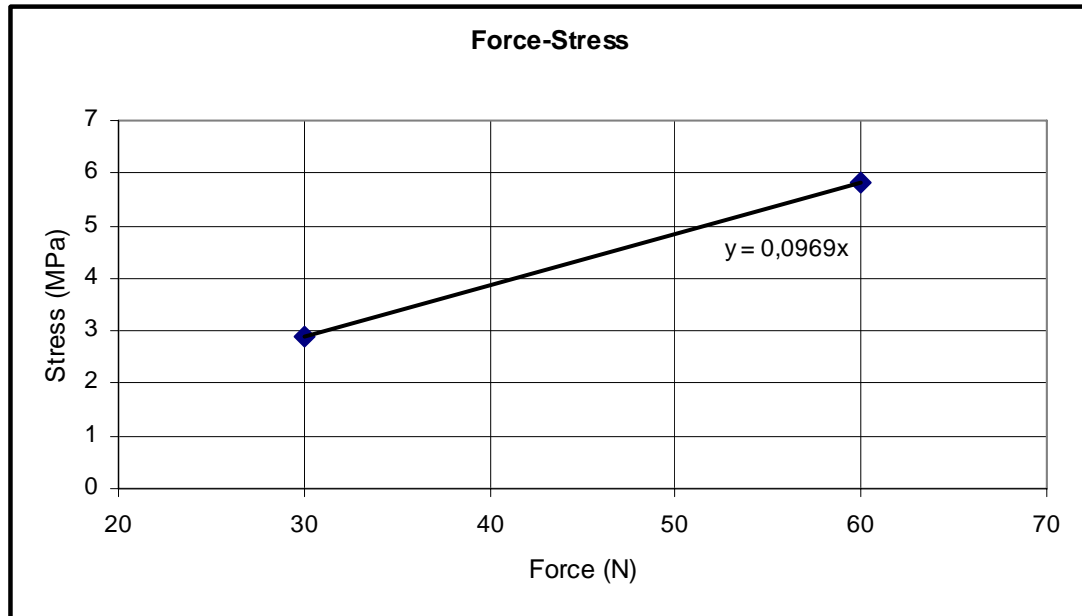


Figure 3.15. Stress-Force Diagram on the Neck Region

Table 3.4. Calculated Stress Values From the Curve

Force (N)	Stress (MPa)
30	2,907
35	3,392
40	3,876
45	4,361
50	4,845
55	5,33
60	5,814

After the finite element analysis, for executing the fatigue test of the linear-low density polyethylene, test specimens are mounted to the fatigue test rig as shown in Figure 3.16.

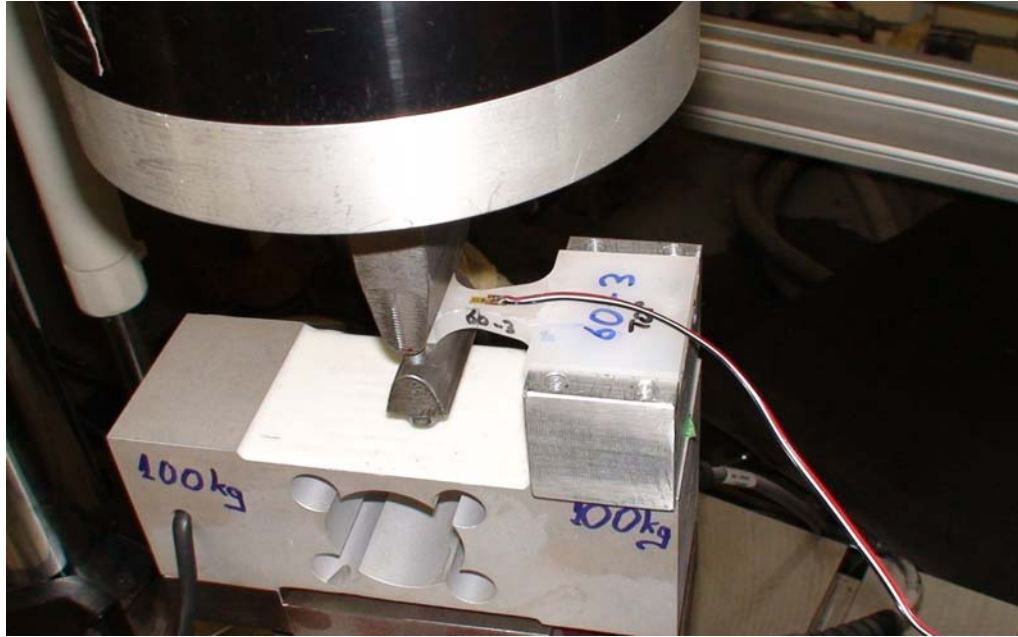


Figure 3.16. Mounting the Test Specimen to the Fatigue Test Rig

With the help of the finite element analysis, the initial force which was applied on the specimens was determined as 60 N. For each load value 3 or 4 test specimens were tested at 30Hz. In the test standard it is mentioned that executing the tests at 30 Hz reduces the test duration [12]. Throughout the tests, it was not seen any change at temperature of the specimens. Strain value was recorded from one specimen for each load condition. Number of the specimen that tested at various load conditions is given in Table 3.5.

Table 3.5. Number of the Specimen tested at various load conditions

Force (N)	Number of Specimen
60	4
55	4
50	4
45	3
40	3

Normally in metal fatigue tests, crack or rupture occurs after a definite cycle and thus the cycle to failure of this material can be determined. However in plastic materials like polyethylene, when the fatigue failure occurs, the plastic material softens and can not carry the same load any more. If the load magnitude is constant, displacement value increases gradually throughout the test. For this reason, fatigue

failure is said to occur when the initial displacement value has been increased by a specified amount. This value is determined as 30% increase of the initial displacement value as mentioned in test standard [12]

For the load conditions given in Table 3.5, throughout the tests, the variation in force, strain and displacement values are given in Table 3.6. These data are given for one specimen at each load condition.

Table 3.6. Variation in Force, Strain and Displacement on Various Load Conditions

		Start			Failure		
		F [N]	Strain [$\mu\text{m/m}$]	Displacement [mm]	F [N]	Strain [$\mu\text{m/m}$]	Displacement [mm]
40 N	Max	40.300	-2332.9	-1.139	40.482	-2456.3	-1.434
	Ave.	-0.2	-4132.6	-	-0.3	-4127.2	-
	Min	-40.366	-5908.0	-2.408	-40.297	-5800.6	-2.682
45N	Max	45.393	660.8	-1.100	44.931	342.5	-0.853
	Ave.	-0.5	-1105.1	-	0.3	-1386.3	-
	Min	-45.406	-2834.7	-2.464	-45.341	-3077.4	-2.299
50 N	Max	50.122	-764.3	-1.032	50.116	-1222.3	-0.823
	Ave.	-0.6	-3029.2	-	0.1	-3828.7	-
	Min	-50.218	-5255.1	-2.455	-50.245	-6378.0	-2.543
55 N	Max	55.653	-191.1	-1.075	55.494	111.4	-0.834
	Ave.	1.2	-2108.9	-	0.5	-2128.5	-
	Min	-55.714	-3969.2	-2.864	-55.599	-4267.9	-3.024
60 N	Max	60.256	743.0	-0.110	60.243	462.9	0.201
	Ave.	0.2	-466.3	-	-0.6	-791.5	-
	Min	-60.205	-1593.3	-2.318	-60.214	-1923.2	-2.517

The variation in force, strain and displacement were obtained graphically for each specimen. It is given in Figure 3.17 and Figure 3.18 for 60 N as an example.

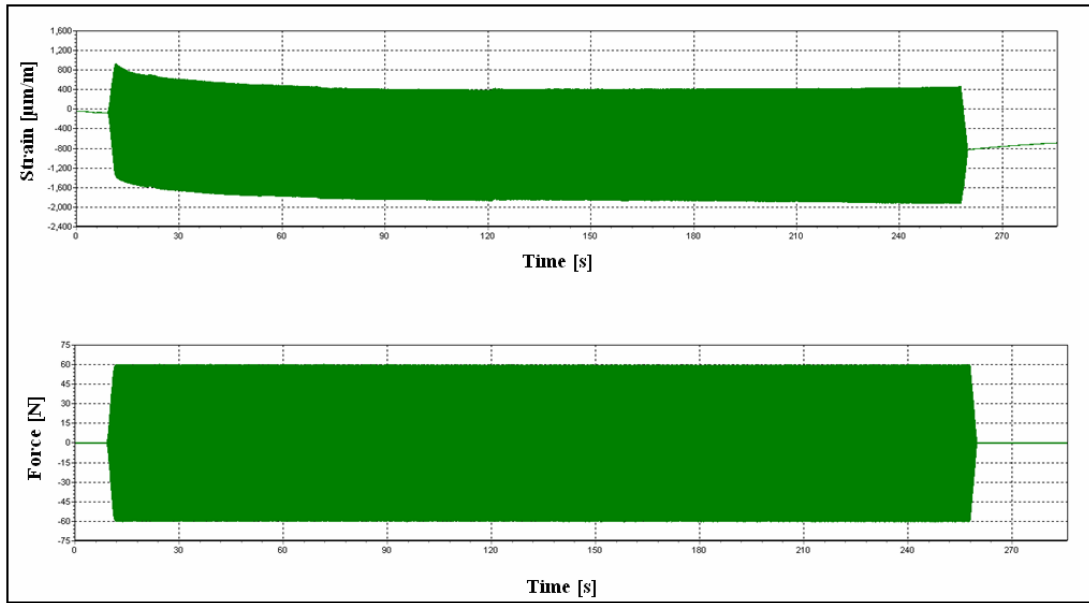


Figure 3.17. Force and Strain Variation at 60 N

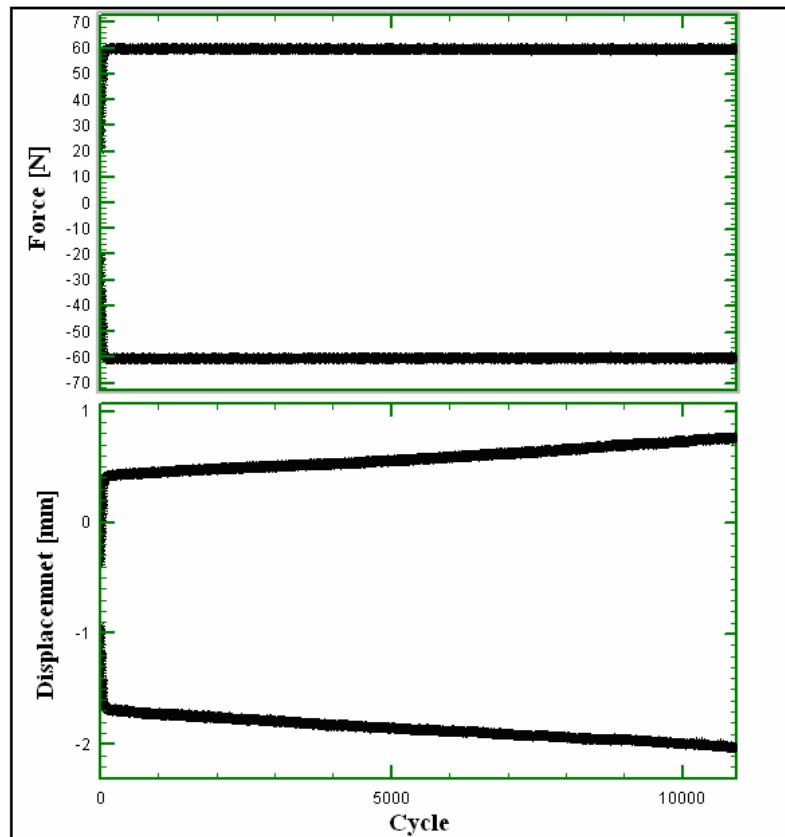


Figure 3.18. Force and Displacement Variation at 60 N

For all specimens cycle to failure and stress on failure area were obtained after the tests and it is given in Table 3.7 [13].

Table 3.7. Test Results

Specimen no.	Force [N]	Stress [Mpa]	Cycle to Failure
MF_01	60,43	5,86	5491
MF_02	60,23	5,84	3619
MF_03	60,17	5,83	7462
MF_04	60,26	5,84	3588
MF_16	55,65	5,39	23693
MF_17	55,13	5,34	10315
MF_18	55,07	5,34	9788
MF_19	55,37	5,36	12660
MF_08	50,44	4,89	32872
MF_11	50,12	4,86	77995
MF_12	50,39	4,88	84051
MF_14	50,13	4,86	51287
MF_21	45,39	4,40	91471
MF_22	45,48	4,41	148764
MF_23	45,24	4,38	317049
MF_25	40,30	3,91	1551737
MF_28	39,88	3,86	5004983
MF_05	40,20	3,89	2230194

According to the Table 3.7 S-N curve of linear-low density of polyethylene was plotted as log-log axis, as shown in Figure 3.19.

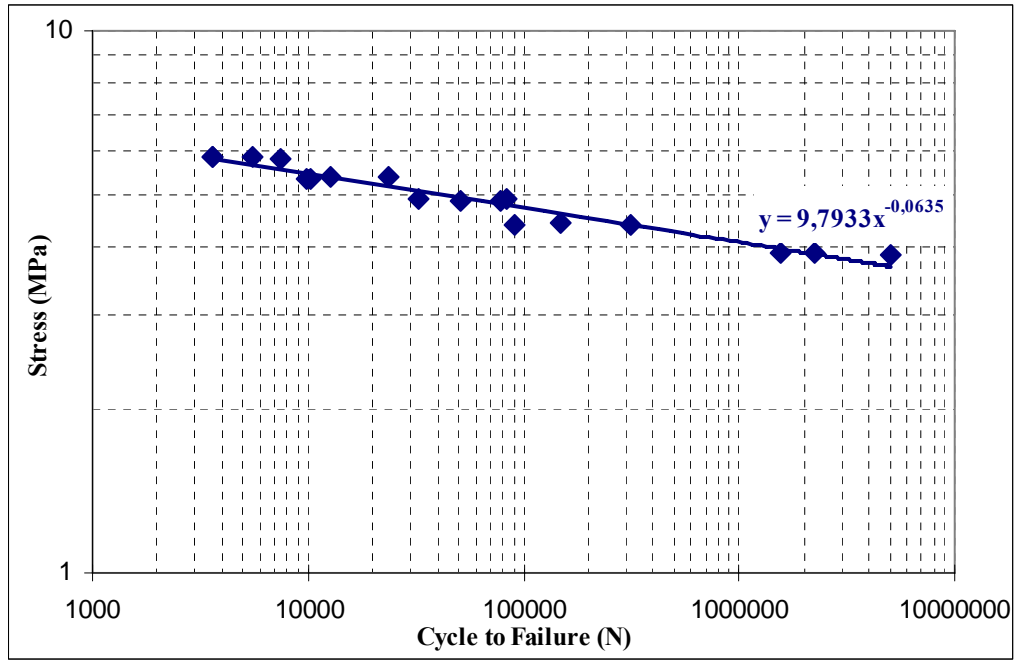


Figure 3.19. S-N Curve of Linear-Low Density of Polyethylene

In general the equation of the S-N curves are expressed like;

$$S = S_0 \cdot (N)^b \quad (4.1)$$

In equation (4.1), generally S_0 is the stress intercept value at $N=1$ and the slope of the curve is often written as b and the value of the slope is approximately -0,1 (see Figure 3.20)

$$b = \frac{\log S - \log S_1}{\log N - \log N_1} \quad (4.2)$$

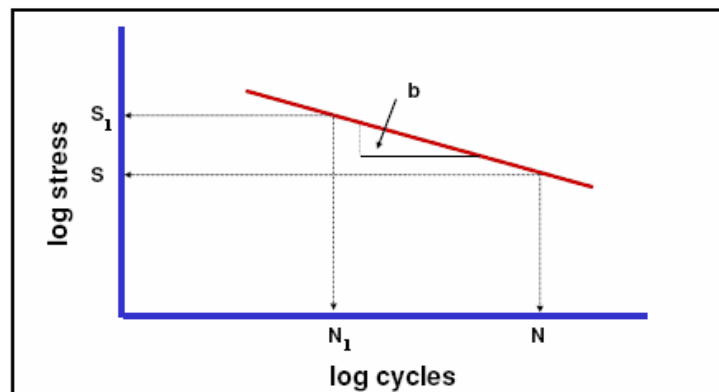


Figure 3.20. The Slope of S-N Curve

As seen in Figure 3.19, the equation of the S-N curve is $S=9,7933.(N)^{-0.0635}$ and the slope of the curve (b) is -0.0635. It can be clearly seen that the obtained S-N curve of LLDPE has similar fatigue characteristics to the basic PE S-N curve, mentioned in section 2.2.2., see Figure 2.11.

4. DISCUSSION AND CONCLUSION

In this study, the intention is to investigate the fatigue behavior of LLDPE which is used as AdBlue tank material in Mercedes-Benz buses. For this purpose, the S-N curve of LLDPE is generated for standard smooth material specimens, (ASTM D671). Standard smooth specimens are flat, un-notched precision-machined with polished surfaces so as to minimize surface roughness effects. The S-N curve of LLDPE provides the baseline fatigue data on a given geometry, loading condition, and material processing for use in subsequent fatigue life and strength analyses. This baseline data can be adjusted to account for realistic component conditions such as notches, size, surface finish, surface treatments, temperature, and various types of loading.

Other than from testing, there is no rational basis for determining these correction factors. The S-N curve for real components, subassemblies, or structures such as AdBlue tanks, represents the true fatigue behavior of production parts/structures including all the aforementioned variables. However, if a design has changed, it is necessary to regenerate the S-N curve to incorporate the change effect. This adds cost and time to the fatigue design process.

Based on the outcome of the studies conducted, another important parameter which is endurance limit has been found. The endurance limit is defined as the stress level below which a material will withstand cyclic stresses indefinitely without failure. As a result, it is obvious that the knowledge of such a stress level is important to designers since it gives them a realistic reference stress on which they can base their design.

Besides endurance limit, the S-N curve of the LLDPE provides other important fatigue parameters about the material. However, in order to calculate the service life in mileage of these AdBlue tanks, the effects such as stress concentrations, surface finishing, temperature etc. at critical locations must be known and according to these factors the obtained S-N curve should be modified.

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AUTOBIOGRAPHY

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