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جامعة بابل كلية هندسة المواد قسم البوليمرات والصناعات البتروكيماوية

Effect of processing parameters on the mechanical properties of HDPE for Industrial industry

بحث مقدم

لقسم البوليمرات والصناعات البتروكيماوية ـ كلية هندسة المواد ـ جامعة بابل
ضمن متطلبات الحصول على درجة البكالوريوس

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Effect of processing parameters on the mechanical properties of HDPE for Industrial industry

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بسم الله الرحمن الرحيم

﴿وَلَمَّا بَلَغَ أَشُدَّهُ وَاسْتَوَى آتَيْنَاهُ حُكْمًا وَعِلْمًا وَكَذَلِكَ نَجْزِي الْمُحْسِنِينَ﴾

صدق الله العظيم

سورة القصص- الآية [١٤]

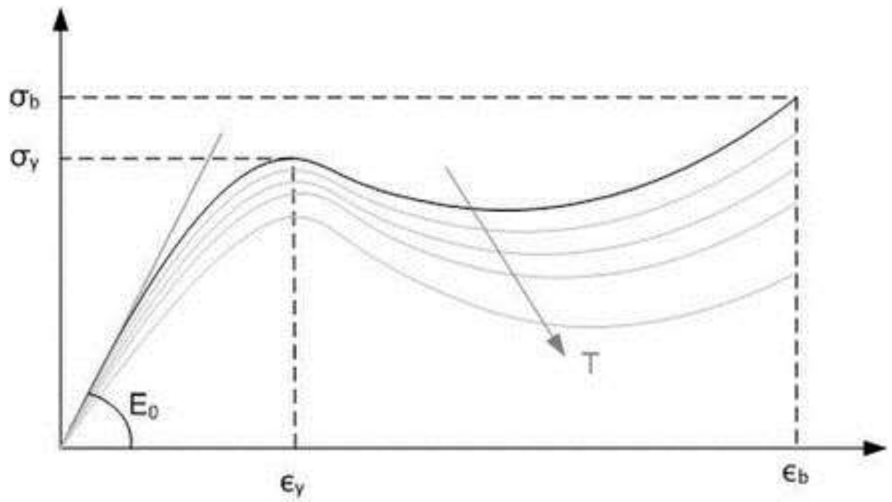
Chapter One

Introduction

1-1Introduction

Engineering thermoplastics are highly crucial for various developments and applications. It is much In demand due to Its low cost, durability, and ease of productivity. It is shapeable In desired dimensions. Among the various thermoplastics, polyether ether ketone is well known as an engineering thermoplastic. It Is possible to obtain desirable mechanical, tribological, chemical, thermal, and electrical engineering properties by deploying polyether ether ketone as the host system. It is the first choice for aerospace, electrical, marine, automotive, and medical applications as a target host system. In this chapter, we report on the various composites and blends of polyether ether ketone, grades, factors affecting its chemical moiety, recycling, and Interfacial issues corelated to its properties, followed by future prospectus**[1]**

Any plastic polymer material that becomes pliable or moldable at a certain elevated temperature and solidifies upon cooling.Most thermoplastics have a high molecular weight. The polymer chains associate by intermolecular forces, **[2]**whichh weaken rapidly with increased temperature, yielding a viscous liquid. In this state, thermoplastics may be reshaped and are typically used to produce parts by various polymer processing techniques such as Injection molding, compression molding, calendering, nd extrusion.**[3]** Thermoplastics differfrom thermosetting polymers (or "thermosets"), which form irreversible chemical bonds during the curing process. Thermosets do not melt when heated, but typically decompose and do not reform upon cooling.



Figer (1-1) Stress-strain graph of a thermoplastic material

Above its glass transition temperature and below its melting point, the physical properties of a thermoplastic change drastically without an associated phase change. Some thermoplastics do not fully crystallize below the glass transition temperature, retaining some or all of their amorphous characteristics. Amorphous and semi-amorphous plastics are used when high optical clarity is necessary, as light Is scattered strongly by crystallites larger than Its wavelength. Amorphous and semi-amorphous plastics are less resistant to chemical attack and environmental stress cracking becausee they lack a crystalline structure**[4]**

1-2 HDPE high density polyethylene

HDPE Is a thermoplastic polymer produced from the monomer ethylene. It is sometimes called "alkathene" or "polythene" when used for HDPE pipes.**[5]** With a high strength-to-density ratio, HDPE is used in the production of plastic bottles, corrosion-resistant piping, geomembranes and plastic lumber. HDPE is commonly recycled, and has the number "2" as its resin identification code.HDPE has SPI resin ID code 2 In 2007, the global HDPE market reached a volume of more than 30 million tons.**[6]**

Table (1-1)Thermophysical properties of high density polyethylene (HDPE) [6]

Density	940 kg/m^3
Melting point	130.8 °C.
Temperature of crystallization	111.9 °C.
Latent heat of fusion	178.6 kJ/kg.
Thermal conductivity	0.44 W/m.°C. at °C.
Specific heat capacity	1330 to 2400 J/kg-K
Specific heat (solid)	1.9 kJ/kg. °C.
Crystallinity	60%

HDPE is known for its high strength-to-density ratio. The density of HDPE ranges from 930 to 970 kg/m^3 The standard method to test plastic density is ISO 1183 (gradient columns), alternatively ISO 1183 part 1 (MVS2PRO density analyzer).[6] Although the density of HDPE is only marginally higher than that of low-density polyethylene, HDPE has little branching, giving it stronger intermolecular forces and tensile strength (38 MPa versus 21 MPa) than LDPE.**[7]** The difference in strength exceeds the difference in density, giving HDPE a higher specific strength.**[8]** It is also harder and more opaque and can withstand somewhat higher temperatures (120 °C/248 °F for short periods). High-density polyethylene, unlike polypropylene, cannot withstand normally required autoclaving conditions. The lack of branching is ensured by an appropriate choice of catalyst (e.g., Ziegler–Natta catalysts) and reaction conditions**[9].**

The traditional method for manufacturing pipe-grade HDPE material from recycled HDPE is the one-step extrusion method.7 With the one-step extrusion method, all the additives are added into the HDPE matrix at the same time, mixed and then reactive extruded. DCP is used as an initiator agent that decomposes into free radicals under thermal treatment to capture the hydrogen molecules of the polyethylene chain. The capture of hydrogen molecules during the macromolecular coupling reaction of radicals forms the desired cross-linked network. Meanwhile, as a free-radical scavenging agent, antioxidants delay the cross-linking of polyolefin to some extent.8 Thus, the efficiency of the antioxidants is reduced when simultaneously used in the presence of free radicals. At the same time, the OIT value decreases too. To meet the requirement that OIT>20 min for the use of plastics in pipes, the amount of antioxidants used is large, which increases the cost**[10]**

This paper presents a new method for processing recycled HDPE into pipe-grade HDPE materials—the two-step extrusion method. In the first step, the initiator and the recycled HDPE matrix are mixed together and processed by reactive extrusion, resulting in the production of the primary HDPE particles. Next, in the second step, the antioxidants are added into the primary HDPE particles to perform the second reactive extrusion. This method can improve the OIT, the ESCR and the elongation at break of the recovered HDPE. The recovered HDPE can be used as extruded pipe, reducing the amount of the antioxidants required, and thereby reducing the cost of HDPE pipe materials. In addition, the effects of the carbon black (CB) in the masterbatch on the OIT were studied. With the increasing CB content, the MFI was nearly constant. When the CB content was <0.8%, the OIT increased as the CB content increased, and when the CB content was >0.8%, the OIT decreased with the CB content. When the CB content was 0.8%, the OIT attained its maximum value**[11]**

Applications of HDPE

HDPE has a wide variety of applications; for applications that fall within the properties of other polymers, the choice to use HDPE is usually economic:**[12]**

- 3D printer filament
- Arena board (puck board)[12]
- Backpacking frames
- Chemical containers
- Chemical-resistant piping
- Coax cable inner insulator
- Conduit protector for electrical or communications cables
- Corrosion protection for steel pipelines
- Electrical and plumbing boxes

Chapter Two
Theoretical part
& Previous Studies

2-1 Extrusion method and types

Extrusion is a process used to create objects of a fixed cross-sectional profile by pushing material through a die of the desired cross-section. Its two main advantages over other manufacturing processes are its ability to create very complex cross-sections; and to work materials that are brittle, because the material encounters only compressive and shear stresses. It also creates excellent surface finish and gives considerable freedom of form in the design process It is divided into types.**[13]**

2-1-1Hot extrusion

Hot extrusion is a hot working process, which means it is done above the material's recrystallization temperature to keep the material from work hardening and to make it easier to push the material through the die. Most hot extrusions are done on horizontal hydraulic presses that range from 230 to 11,000 metric tons (250 to 12,130 short tons). Pressures range from 30 to 700 MPa (4,400 to 101,500 psi), therefore lubrication is required, which can be oil or graphite for lower temperature extrusions, or glass powder for higher temperature extrusions. The biggest disadvantage of this process is its cost for machinery and its upkeep.**[13]**

2-1-2 Cold extrusion

Cold extrusion is done at room temperature or near room temperature. The advantages of this over hot extrusion are the lack of oxidation, higher strength due to cold working, closer tolerances, better surface finish, and fast extrusion speeds if the material is subject to hot shortness.**[14]**

2-1-3 Warm extrusion

In March 1956, a US patent was filed for "process for warm extrusion of metal". Patent US3156043 A outlines that a number of important advantages can be achieved with warm extrusion of both ferrous and non-ferrous metals and alloys if a billet to be extruded is changed in its physical properties in response to physical forces by being heated to a temperature below the critical melting point.[3] Warm extrusion is done above room temperature, but below the recrystallization temperature of the material the temperatures ranges from 800 to 1800 °F (424 to 975 °C). It is usually used to achieve the proper balance of required forces, ductility and final extrusion properties**[15]**

2-2 Previous studies

1. **In 2010 [16]** study researcher M. Bernard at.th. investigates the effects that processing parameters, including temperature and speed, have on The mechanical properties of kenaf fibre plastic composite.Dynamic mechanical analysis (DMA) was carried out to examine the mate-Rial properties.The tensile properties of PP/kenaf compos-Ite increased by 10% after the addition of unidirectional kenaf fibre (UKF).The newly invented compression moulding machine illustrates aNew trend in processing parameters of long kenaf fibre plastic composite.The fracture surface of
2. **In 2021 [17]** Mejia, E.;al. th The fabricated samples were studied HDPE using uniaxial tensile Testing to determine their mechanical performance. Furthermore, the microstructure of samples was Analyzed using different characterization techniques. Compression-molded specimens recordedhigher degree of crystallinity (DC) using two different characterization techniques such as differential Scanning calorimetry (DSC) and X-ray diffraction (XRD). With this information, critical processing Factors were determined, and a general structure
3. **In 2011 [18]** S. Kamaruddin".Present investigates the feasibility of the substitution of recycled plastic with virgin plastic.High Density Polyethylene , and the process parameters were optimized to yield optimum tensile, compressive and flexural strengths. The effect of selected injection moulding process parameters and their optimal settings have been obtained using Taguchi method. The estimated optimal values of mechanical properties e.g. HDPE are 19.900 MPa, 1.228 MPa and 75.127 MPa respectively. These properties differ only by less than 3%from those for the virgin HDPE. Results of investigation, establish that the recycled HDPE can be feasibly substituted for the virgin HDPE as the mechanical properties of recycled HDPE were found close to the virgin HDPE.

Chapter Three

Experimental Part

Preparation of samples

Samples of a material HDPE were manufactured using a device for the abdomen during the screw Sorpm temperature.200 C°Samples were manufactured for different conditions

1. Extrusion samples 2 by 200 tensile, shock and bending tests.

2. Samples are made in 2 extruders, then pressed and wrapped in aluminum and placed in a heat press with a pressure of 1000s at room temperature. Tensile, shock and bending tests were conducted for the samples.

Hardness and density were measured for all samples.

3-1 DSC device

is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. Additionally, the reference sample must be stable, of high purity, and must not experience much change across the temperature scan. Typically, reference standards have been metals such as indium, tin, bismuth, and lead but other standards such as polyethylene and fatty acids have been proposed to study polymers and organic compounds.respectively.**[19]**

Figer (3-1) DSC device

3-2 FTIR device

is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid, or gas. An FTIR spectrometer simultaneously collects high-resolution spectral data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time.**[20]**

Figer (3-2) FTIR device

3-3 Tensile device

is a fundamental materials science and engineering test in which a sample is subjected to a controlled tension until failure. Properties that are directly measured via a tensile test are ultimate tensile strength, breaking strength, maximum elongation and reduction in area.[2] From these measurements the following properties can also be determined: Young's modulus, Poisson's ratio, yield strength, and strain-hardening characteristics.[3] Uniaxial tensile testing is the most commonly used for obtaining the mechanical characteristics of isotropic materials. Some materials use biaxial tensile testing. The main difference between these testing machines being how load is applied on the materials.**[21]**

Figer (3-3) Tensile devicetg

3-4 Bending device

is a forming machine tool (DIN 8586). Its purpose is to assemble a bend on a workpiece. A bend is manufactured by using a bending tool during a linear or rotating move. The detailed classification can be done with the help of the kinematics **[22]**

Figer (3-4) Bending device

3-5 Charpy testa device

In materials science, the Charpy impact test, also known as the Charpy V-notch test, is a standardized high strain rate test which determines the amount of energy absorbed by a material during fracture. Absorbed energy is a measure of the material's notch toughness. It is widely used in industry, since it is easy to prepare and conduct and results can be obtained quickly and cheaply. A disadvantage is that some results are only comparative.[1] The test was pivotal in understanding the fracture problems of ships during World War II **[23]**

Figure (3-5) Charpy testa device

3-6 Densometer device

Nuclear densitometry is a technique used in civil construction and the petroleum industry, as well as for mining and archaeology purposes, to measure the density and inner structure of the test material. The processes uses a nuclear density gauge, which consists of a radiation source that emits particles and a sensor that counts the received particles that are either reflected by the test material or pass through it. By calculating the percentage of particles that return to the sensor, the gauge can be calibrated to measure the density**. [24]**

Figure (3-6) Densometer device

3-7, Shore D Durometer device

The Shore durometer is a device for measuring the hardness of a material, typically of polymers.Two inline skate wheels with different durometer – 85A and 83A Digital Shore hardness tester Higher numbers on the scale indicate a greater resistance to indentation and thus harder materials. Lower numbers indicate less resistance and softer materials.The term is also used to describe a material's rating on the scale, as in an object having a "'Shore durometer' of 90."The scale was defined by Albert Ferdinand Shore, who developed a suitable device to measure hardness in the 1920s. It was neither the first hardness tester nor the first to be called a durometer (ISV duro- and -meter; attested since the 19th century), but today that name usually refers to Shore hardness; other devices use other measures, which return corresponding results, such as for Rockwell hardness. **[25]**

Figer (3-7) Shore D Durometer device

3-8 extrusion device

extrusion machine is a device which pushes or pulls a material through a shaped die to form a continuous length of product with a preset cross section. The extrusion process is used to produce a large number of commercial products which include steel or copper wire, plastic tubing, plastic sheets, and many food types. Extrusion as a manufacturing process offers many benefits such as the wide range of complex cross sections possible and the ability to form brittle materials. Depending on the material used, an extrusion machine may form the material cold or hot with some types of materials being completely melted prior to extrusion.**[26]**

Figer (3-8) extrusion device

2-9 Heat press device

A hot hydraulic press or a heat press machine is a device that offers easy press and operation. Heat transfer changes the internal energy of both systems involved according to the First Law of Thermodynamics. The heating-up speed is selectable according to different products. The titanium indenter ensures an even temperature, fast heat-up, and long service life. The pressure head is designed to be adjustable to ensure even pressure on the component. The temperature is controlled for clear precision. Digital pressure gauge with preset pressure range.**[27]**

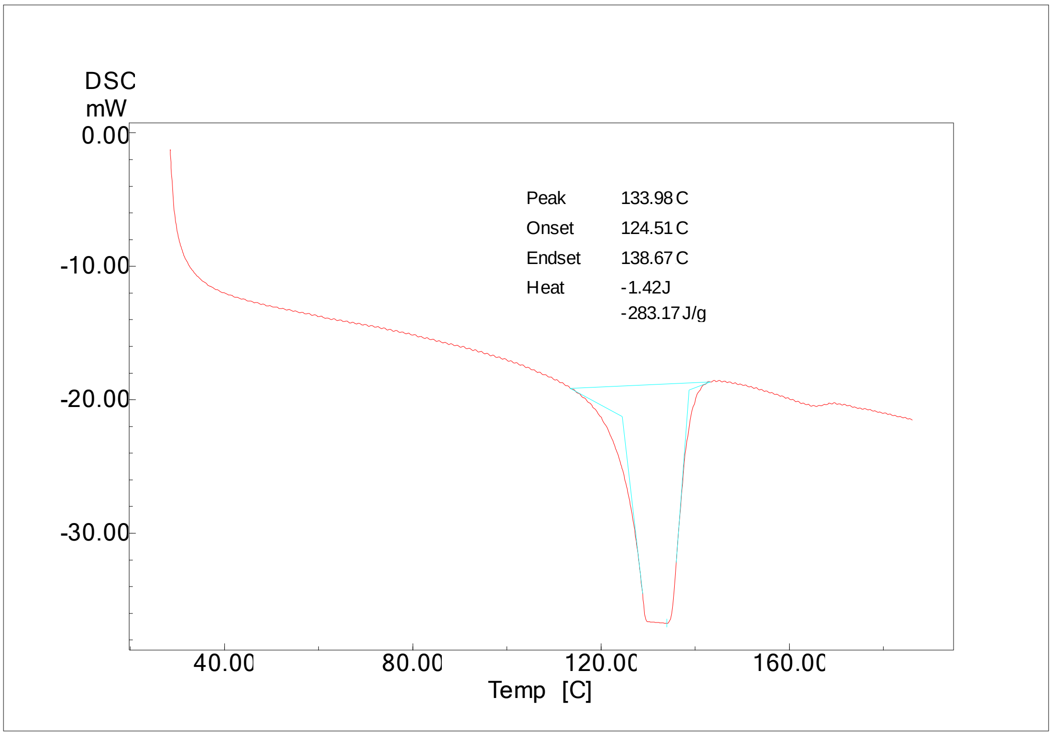
Figer (3-9) Heat press device

Chapter Four

Results & Discussion

4-1 DSC device

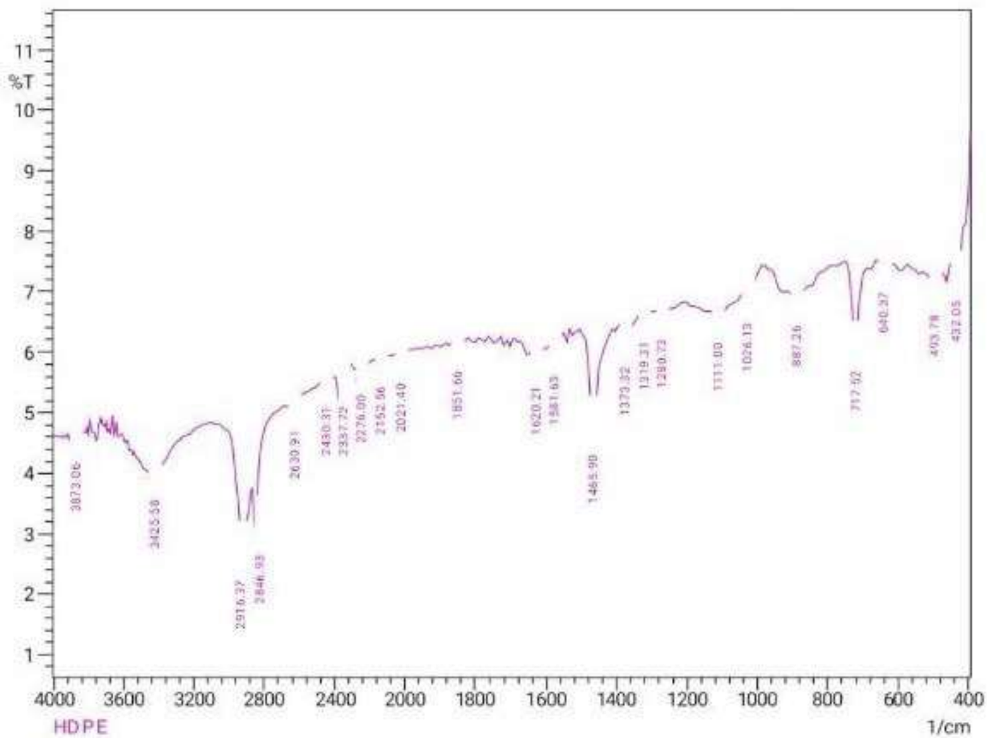
A powder was taken from a sample of high-density polyethylene and heated by a differential calorimeter (DSC) device, at a heating rate of 10 ° C / min. As shown in the figure, the melting point of the material was approximately 135 ° C.



Figer (4-1) laboratory

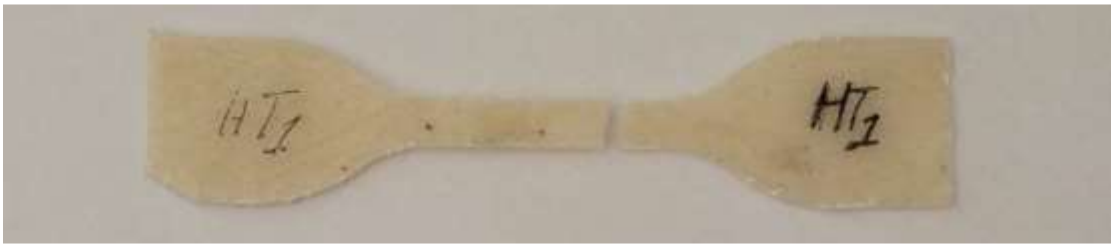
4-2 FTIR device

examined the analysis of the Fourier transform with infrared waves. A chemical analysis of the sample was performed using the FTIR device by taking a powder sample from the sample. The chemical analysis of high-density polyethylene (HDPE) was obtained.

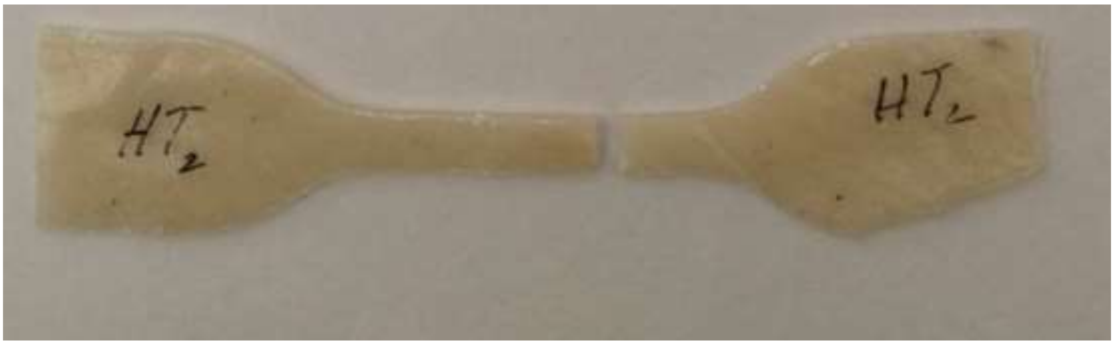


Figer (4-2) laboratory

4-3 Tensile Test



HT1



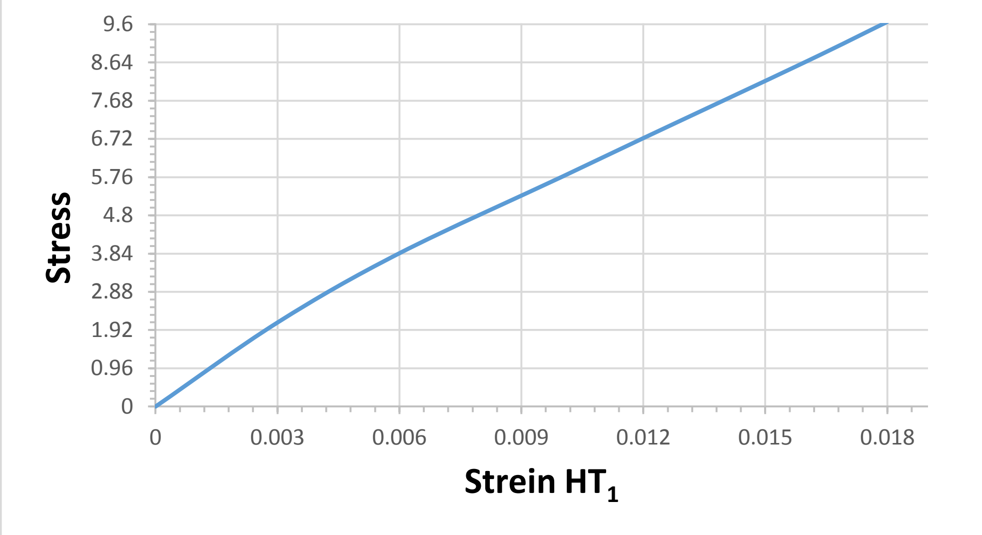
HT2

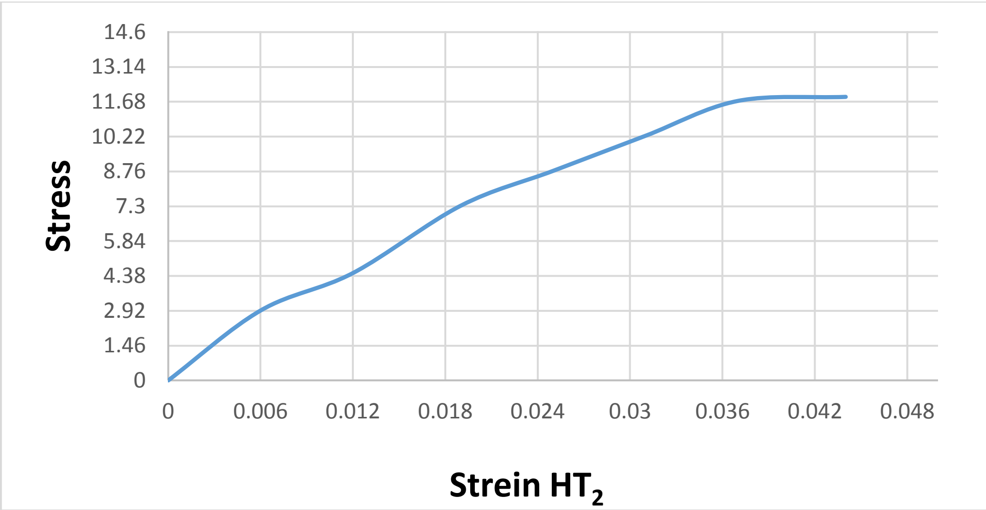
HT3

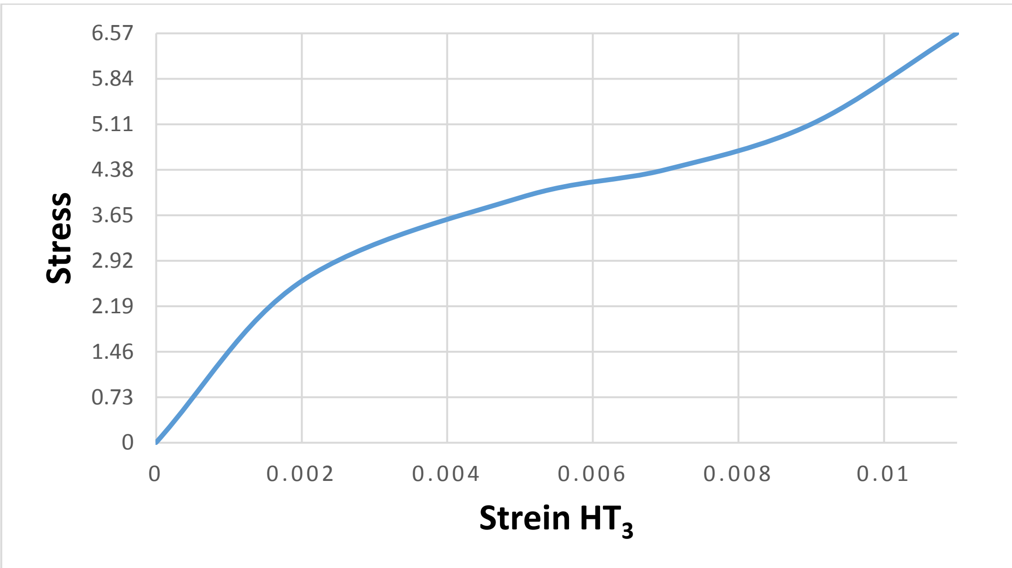
HT4

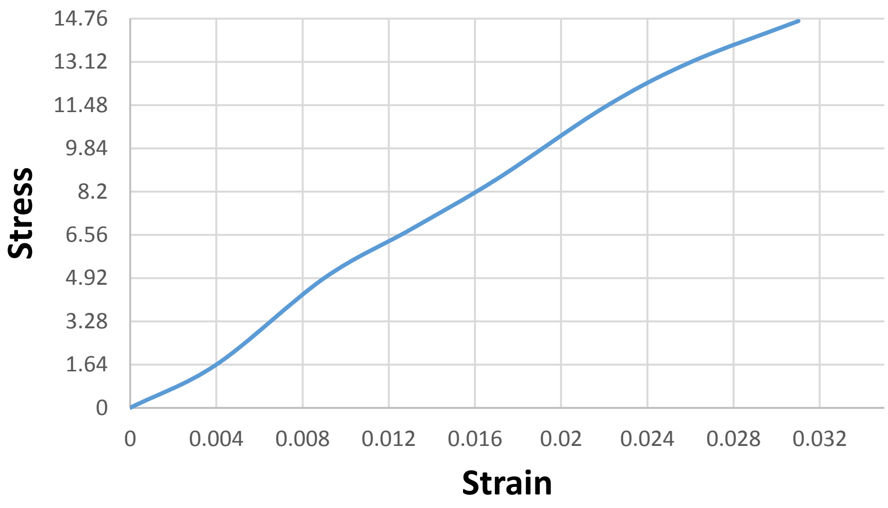
Figer (4-3) samples of tensile test

- ❖ to achieve that all samples have a brittle fracture


9.6
8.64
7.68
6.72
5.76
4.8
3.84
2.88
1.92
0.96
0
Stress
0
0.003
0.006
0.009
0.012
0.015
0.018
Strein HT1


14.6
13.14
11.68
10.22
8.76
7.3
5.84
4.38
2.92
1.46
0
Stress
0
0.006
0.012
0.018
0.024
0.03
0.036
0.042
0.048
Strein HT2


6.57
5.84
5.11
4.38
3.65
2.92
2.19
1.46
0.73
0
Stress
0
0.002
0.004
0.006
0.008
0.01
Strein HT3

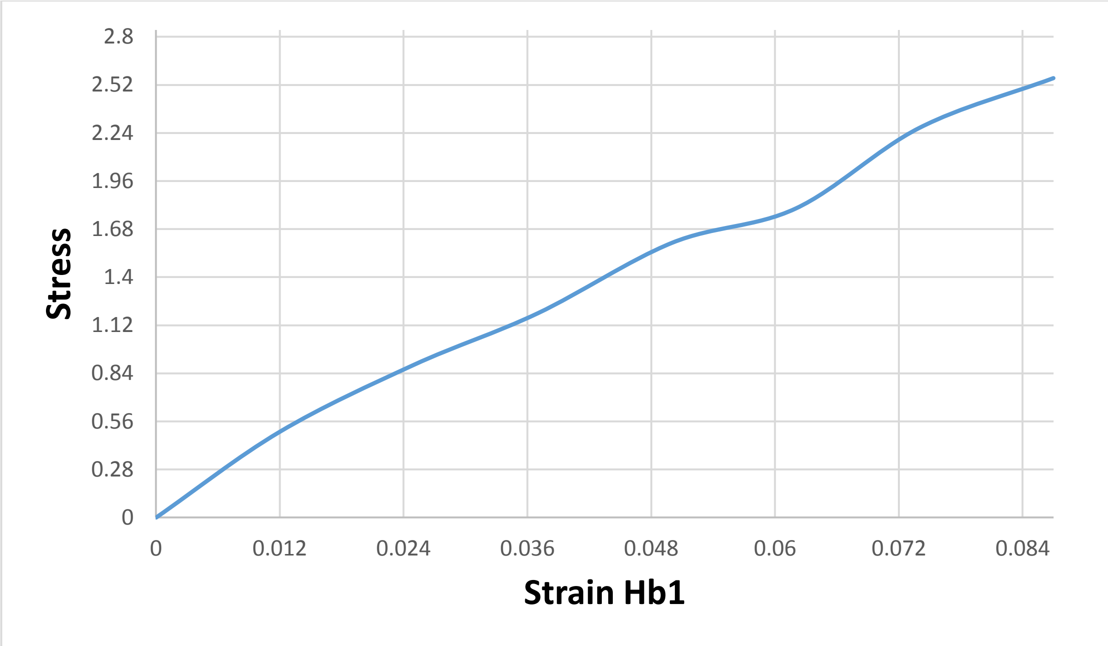


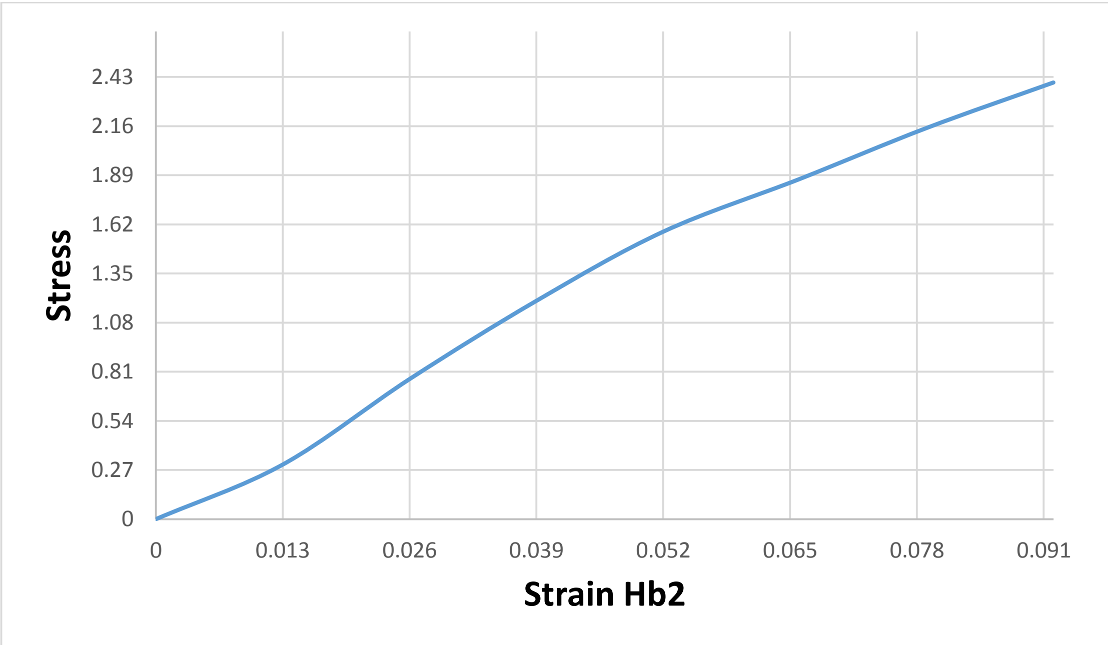
Elastic modulus=$tan\theta = \frac{\delta_2 - \delta_1}{\varepsilon_2 - \varepsilon_1}$

	calculated values	laboratory values	Tensile strength
HT1	0.420	0.58	9.6
HT2	0.34	0.29	11.68
HT3	0.698	0.58	6.57
HT4	0.46	0.57	14.76

The tensile strength of the samples HT1, HT2, HT3, HT4 was compared, and the sample HT4 had a higher tensile strength, as it took a longer period until the fracture occurred, and this gives an indication that the annealing method in this sample is the best

4-4 Bending Test





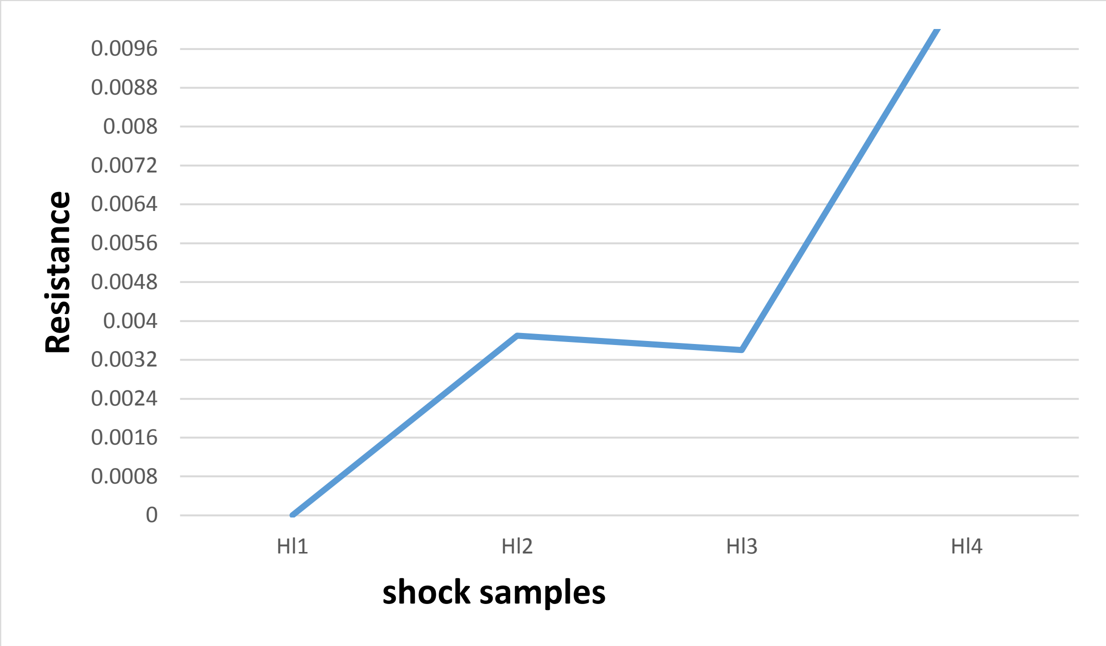
The highest impact resistance was obtained through the HI4 sample that was extruded and pressed.

	laboratory modulus	calculated modulus	Bending strength
Hb1	0.0013	0.03	2.43
Hb2	0.018	0.03	2.61
Hb3	/	0.02	1.75
Hb4	/	0.01	0.29

We write that the sample Hb2 is more resistant to bending so that it did not break quickly because the conditions of its manufacture were more resistant to bending

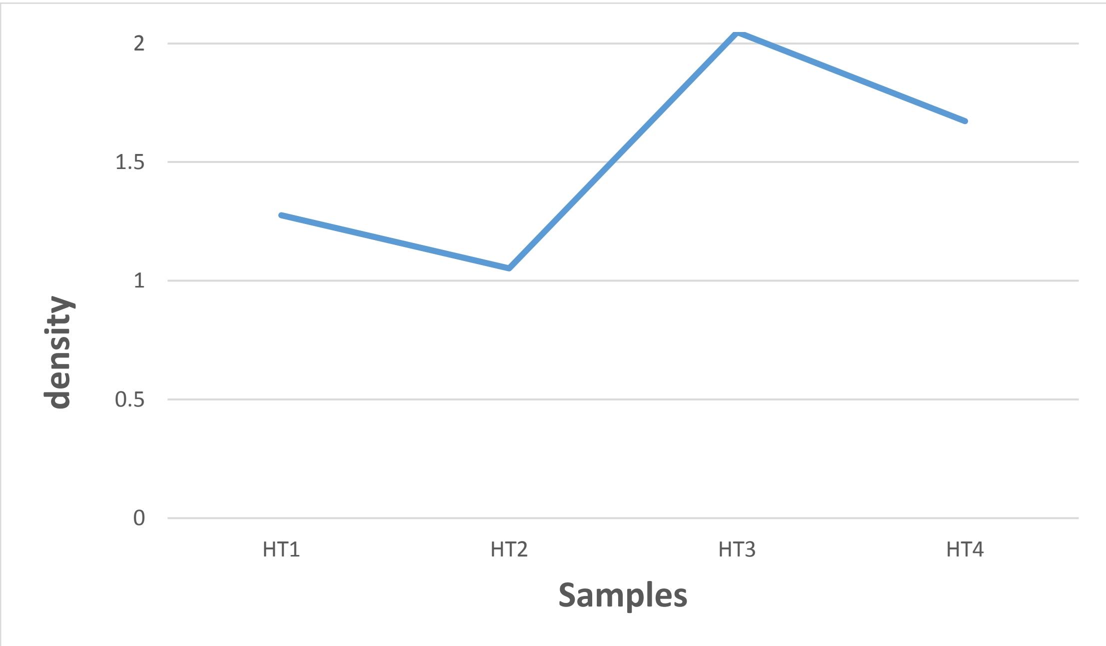
4-5 Charpy testa device

$$impact\ resistance = \frac{shock\ energy}{sectional\ area}$$

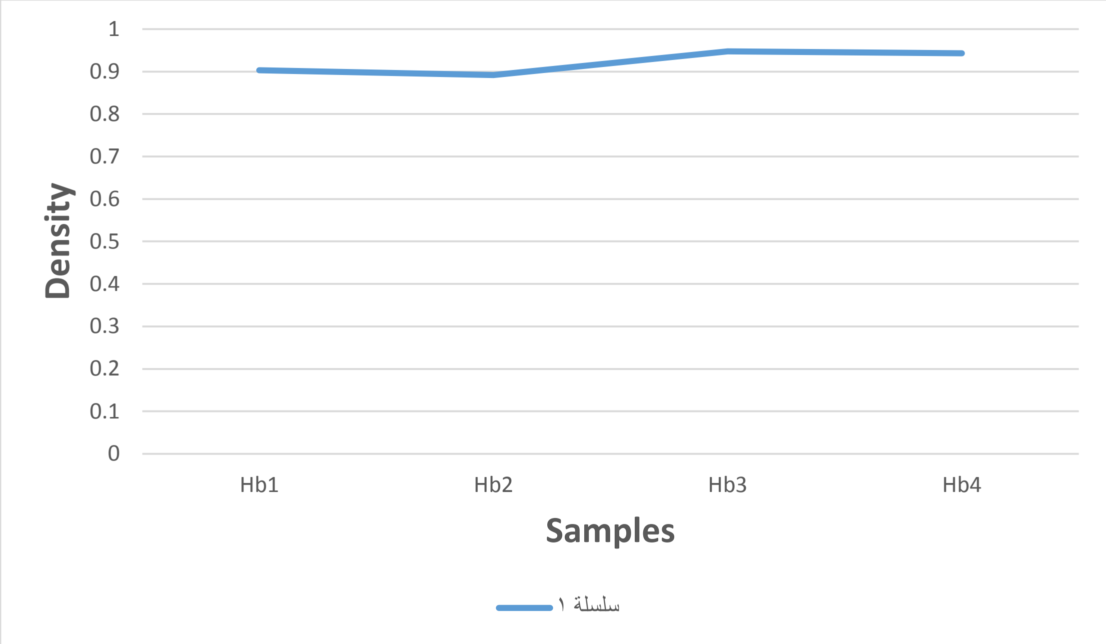


We note that sample HI4 has a higher shock resistance than the rest of the samples, and that sample HI1 is less resistant to shock.

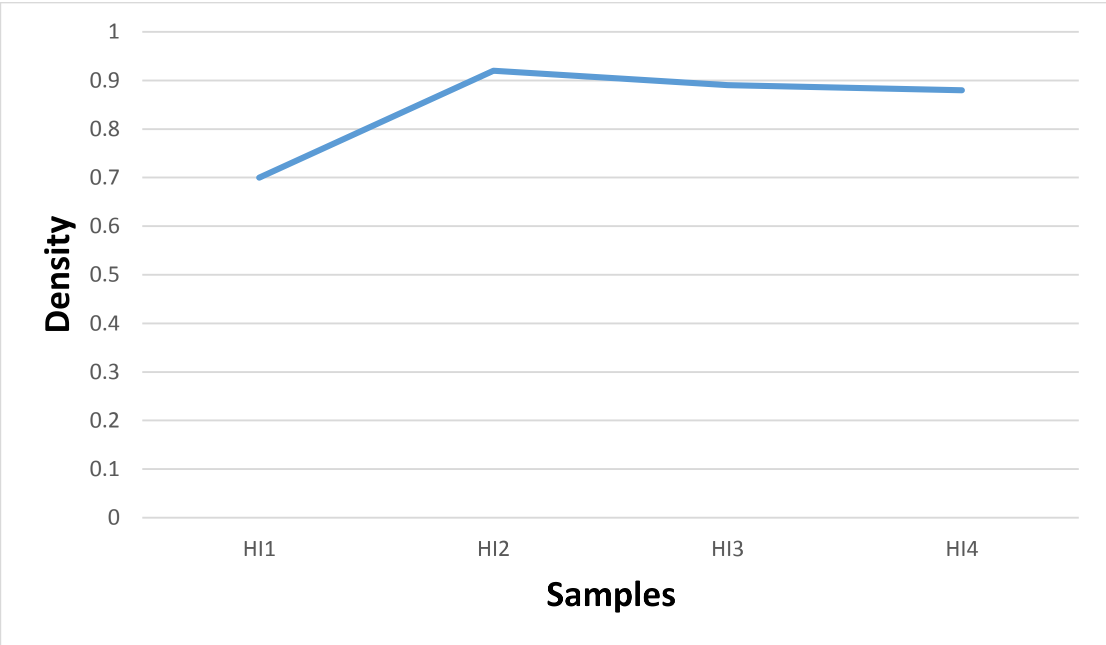
4-6 Densometer device



The density test was carried out for the tensile samples, and the sample HT3 had a higher density than the rest of the samples.

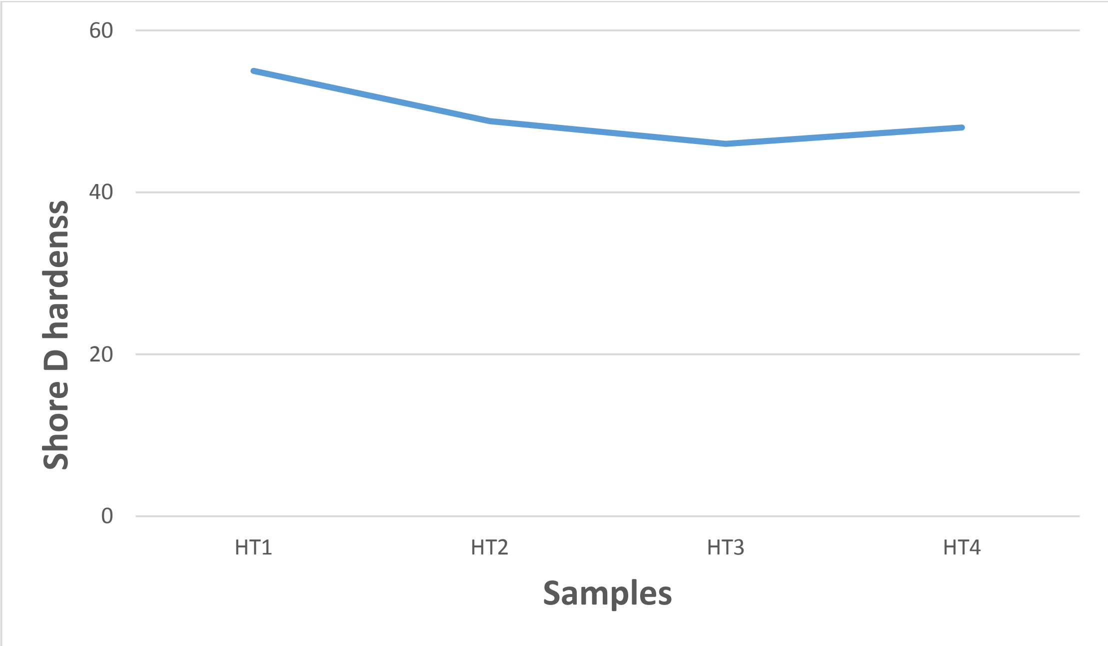


The density test was carried out for the bending samples, and the sample Hb3 had a higher density than the rest of the samples

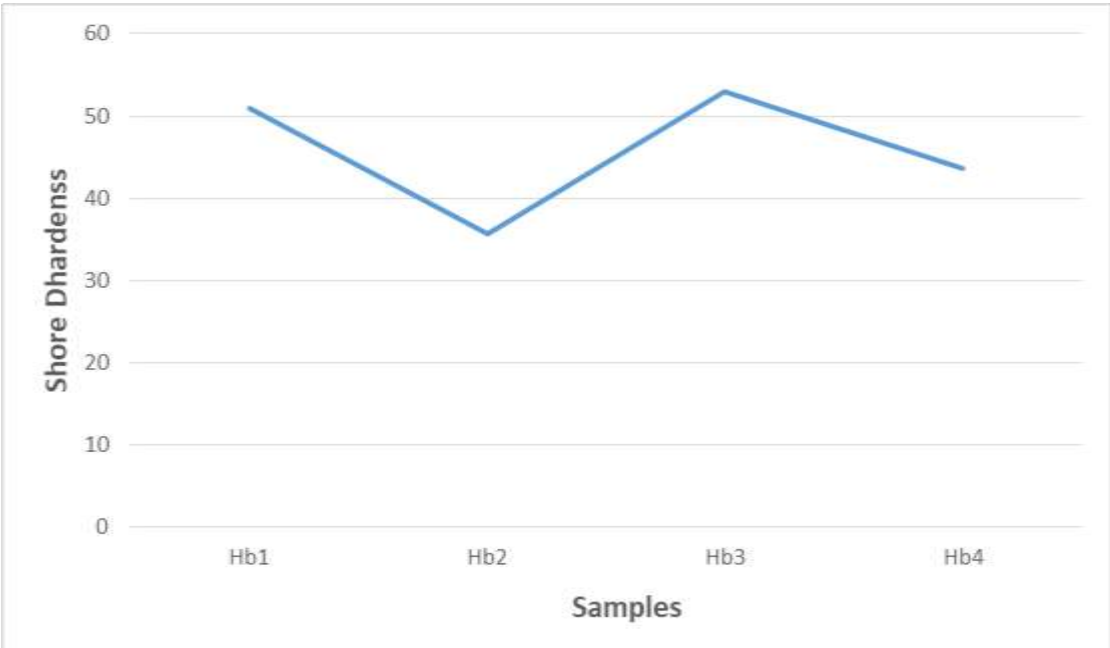


The density test was carried out for the shock samples, and the Hl2 sample had a higher density than the rest of the samples

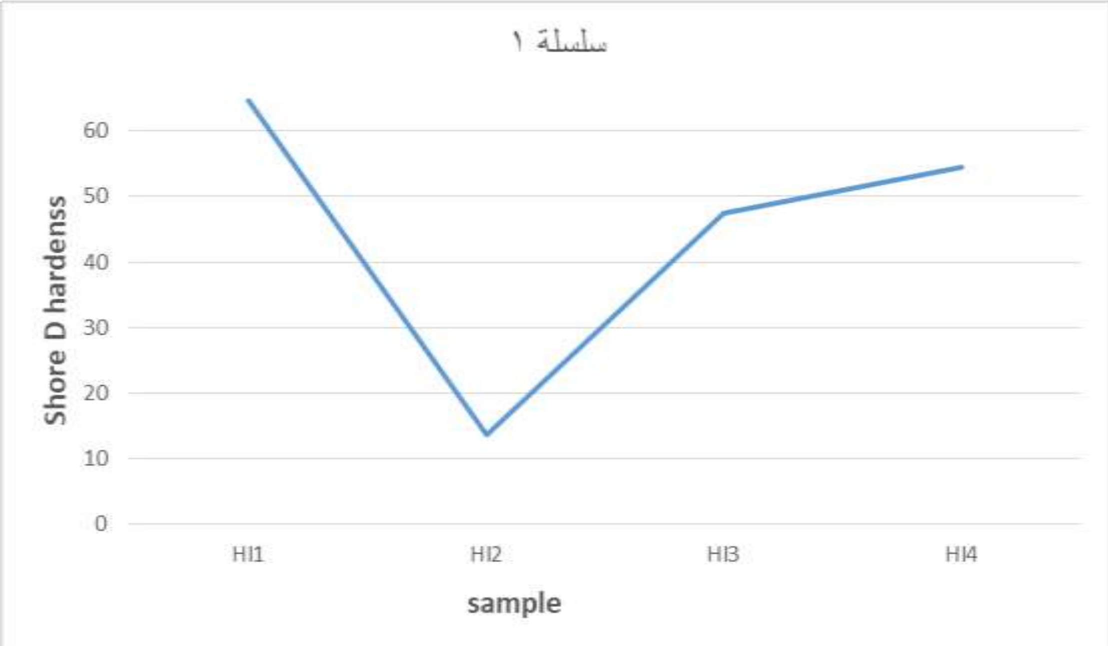
4-7 Shore D Durometer device



After conducting the hardness test, we notice that the sample HT2 has the highest hardness and the sample HT1 has the lowest hardness



The hardness test was conducted for the bending samples as well, and the sample Hb3 was the most hardness and the sample Hb2 was the least hardness



The hardness test was carried out for the shock samples, and the sample HI1 was the most hard and the sample HI2 was the least hard.

CONCLUSIONS

1. The highest modulus of elasticity was obtained from the tensile samples, and the sample HT3, which was extruded and placed in the piston, had the highest modulus of elasticity. As for the tensile strength, the sample HT4 was the highest tensile strength, and it was also extruded and put in the piston
2. The bending test was performed The sample Hb2 had the highest modulus of elasticity, as this sample was extruded only at a temperature of 200 ° C. As for the tensile strength, the sample Hb2 also had the highest bending resistance
3. Through the HI4 sample that has been extruded and pressed
4. And we conducted a density test for each of the tensile, bending and shock samples, and we concluded that the sample HT3 and Hb2 have the highest density
5. As for the hardness test, we also conducted a test for tensile, bending and shock samples, and the HI3 sample was the most hard

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