UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: Effect of Nano Calcium Carbonate On Epoxy Composite Reinforced Recycle Rubber

SESI PENGAJIAN: 2010/11 Semester 2

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Author's Name	:	Wan Mohd Hafiz Bin Wan Harun
Date	:	

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the Degree in Bachelor of Manufacturing Engineering (Engineering Materials). The member of the supervisory committee is as follow:

(Signature of Supervisor)

………………………………
(Official Stamp of Supervisor)

(Signature of Co-Supervisor)

………………………………
(Official Stamp of Co-Supervisor)

ABSTRAK

Tujuan utama projek ini adalah untuk mengetahui pengaruh kalsium karbonat nano pada sifat mekanik dari kitar semula getah epoksi dikeraskan komposit. peratusan berat yang berbeza nano kalsium karbonat akan memberikan sifat mekanik berbeza dengan komposit epoxy. Penguatan elastomer oleh zarahan suapan semua sebahagian besar berdasarkan interaksi fizikal antara suapan dan matriks getah.Dalam beberapa tahun terakhir, nano-saiz zarah organik telah menarik perhatian sebagai agen ketangguhan baru, kerana bahan yang dihasilkan menunjukkan sifat mekanik dan terma unik yang tinggi. serbuk Nano-$CaCO_3$ adalah yang termurah yang tersedia secara komersial, dan mempunyai keuntungan tambahan dari aspek nisbah yang rendah dan kawasan permukaan besar. Projek ini bermula daripada mempersiapkan sampel dengan menambah resin epoxy dengan kitar semula getah dengan nisbah 1: 0,4. Bahan tersebut aduk dengan menggunakan penguli mekanik di 500rpm kelajuan sampai tercampur. Setelah itu serbuk $CaCO_3$ nano akan ditambah dengan 5 peratus berat yang berbeza (0%, 2%, 4%, 6%, dan 8%) .. Serbuk nano-$CaCO_3$ ditambah dan dicampur dengan menggunakan penguli mekanik kilang pada kelajuan 500 rpm selama 5 minit. Kemudian ditambah pembesar epoxy dengan cara yang sama, pada kelajuan 500 rpm selama 5 minit. Setelah campuran siap, campuran ini akan dituangkan ke dalam cetakan dan kemudian tunggu sekitar 5 jam sehingga sembuh. Kemudian sampel akan sembuh dalam tungku dan dipanaskan pada 140 ° C selama 1 jam. Setelah menyembuhkan, uji mekanikal dilakukan dan permukaan retak spesimen yang diperiksa di bawah Mikroskop Elektron (SEM). Setelah semua ujian di buat keputusan menunjukkan pertambahan kekuatan pada bahan epoxy komposit dengan peningkatan kadar nano $CaCo_3$.

ABSTRACT

The main purpose for this project is to investigate the effect of nano calcium carbonate on the mechanical properties of epoxy composite reinforced recycle rubber. Different weight percentage of nano calcium carbonate will give different mechanical properties to the epoxy composite. The reinforcement of elastomers by particulate fillers is achieved largely on the basis of physical interactions between the filler and the rubber matrix. In recent years, nano-size organic particles have attracted considerable attention as new toughening agents, because the resulting materials exhibit uniquely high thermal and mechanical properties. Nano-$CaCO_3$ powder is the cheapest commercially available, and has the additional advantages of a low aspect ratio and a large surface area. This project is start from preparing of sample by adding the epoxy resin with recycle rubber with ratio 1 : 0.4. The material were stir using mechanical stirrer at speed 500rpm until it mixed. After that nano $CaCo_3$ powder will added with 5 different weight percent (0%, 2%, 4%, 6%, and 8%). The nano-$CaCO_3$ powder was added and mixed using the mechanical stirrer mill at speed 500 rpm for another 5 min. Then the epoxy hardener were added in the same manner, at speed 500 rpm for 5 min. After the mixture is prepared, this mixture will poured into the mould and then wait about 5 hour until it cured. Then the sample will cured inside the furnace and be heated at 140 °C for 1 hour . After curing, mechanical tests were performed and the fractured surfaces of the specimens were examined under Scanning Electron Microscopy (SEM). The results show that as we increased the nano $CaCo_3$ to the epoxy composite, the strength of mechanical properties are been improved.

ACKNOWLEDGEMENT

First of all, Alhamdullilah praise to God because I have finished my final year project 1. I also gratefully acknowledge to my PSM co-supervisor, Cik Nooririnah Omar for his encouragement, fully support, by providing me enormous guidance and idea for my research project. Also for my supervisor, Cik Siti Rahmah Shamsuri for his guidance, advice and motivated me to complete my final year project 1 successfully.

Finally yet importantly, thankfulness to my parent’s Mr. Wan Harun B. Wan Abdul Rahman and Mrs. Wan Kelthom bte Wan Ali who encouraged me all along and helped me through the difficult times even I am far away from them. For my entire group members’ final year project, thanks to all of you in supported and gives the same idea to me. Thanks to Allah, thanks for all. Besides those mentioned, I also would like to express my gratitude with highly appreciation and dedication to my family because without them, I won’t be here to complete my study in Universiti Teknikal Malaysia, Melaka.

DEDICATION

This report is dedicated to my parents, En Wan Harun Wan Abdul Rahman and Pn Wan Kelthom Wan Ali, my siblings, my co-supervisor Cik Nooririnah Omar and, my supervisor, Cik Siti Rahmah Samsuri, and all my friends.

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LIST OF ABBREVIATIONS

%	-	Percentage
^{o}C	-	Degree Celcius
ASTM	-	American Society for Testing and Materials
T	-	Temperature
t	-	Time
mins	-	Minutes
$CaCO_3$	-	Calcium Carbonate
GCC	-	Ground Calcium Carbonate
PCC	-	Precipitated Calcium Carbonate
BR	-	Butadiene Rubber
NBR	-	Nitrile Butadiene Rubber
SBR	-	Styrene Butadiene Rubber
SMI	-	Specialty Minerals Inc.
CPE	-	Chlorinated Polyethylene
CAN	-	Acrylonitrile
XNBR	-	Carboxylated Nitrile
ABS	-	acrylonitrile-butadiene-styrene resin

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Epoxides are the most widely used and studied thermosetting materials, used in industrial applications such as coatings, matrices for composites, adhesives, and encapsulating materials. Neat epoxides, however, have a very low crack growth resistance and are among the most brittle polymeric materials.

Addition of a second dispersed rubbery particulate phase with several microns, i.e. rubber modification, has been successfully applied to overcome the brittleness of many epoxides over the past 25 years. There are two approaches for rubber modification of epoxides. The first approach, which has been in use since 1970, is based on using reactive liquid elastomers . The reactive liquid rubber used in this method is first dissolved in the epoxy. After addition of a curing agent, as the resin begins to cure and the molecular weight rises, the rubber precipitates out and forms the second phase particles. Volume fraction and size of the rubber domains are controlled by the degree of compatibility of two phases and the kinetics of gelation. Butadiene-acrylonitrile based rubbers are the principal liquid elastomers used for toughening of epoxies. The second approach for rubber modification of epoxies, which is a relatively recent development, is based on using pre-formed rubber particles instead of reactive liquid elastomer. The advantage of this approach is that particle size and volume fraction of the second phase are usually not varied by the resin type and by processing conditions. Compatibility with the epoxy resin and adhesion to the matrix can be achieved via chemistry of the surfaces of the particles

Nano-calcium carbonate (nano-$CaCO_3$) is one of the most common nano-sized fillers used in preparation of nano composites. There are two general classes of $CaCO_3$, the ground natural $CaCO_3$ and the synthetic or precipitated $CaCO_3$. The ground natural $CaCO_3$ are generally larger in size and have a much broader particles size distribution than the precipitated $CaCO_3$. All the $CaCO_3$ were surface treated with a fatty acid (Mathur, 1986).

As an important basic material, Calcium Carbonate is widely applied in traditional industry and new industry such as Plastics, Rubber, Papermaking, Printing Ink, Coatings and Paints, and Building Materials. According to different productive methods, Calcium Carbonate includes Ground Calcium Carbonate (GCC) produced by physical method, and Precipitated Calcium Carbonate (PCC) produced by chemistry method, and precipitated calcium carbonate (PCC) produced by chemistry method. It is a super ultra fine and very narrow particle size distribution precipitated calcium carbonate specially formulated as functional filler and extender in rubber products.

1.2 Problem Statement

Investigate the effects of different weight percent of nano $CaCO_3$ on mechanical properties of the epoxy composite toughened recycle rubber that can help the researches to determine the mechanical property that can replace for the bulk material in nowadays industry and engineering design purpose.

1.3 Objective

The objectives of this project are:-

(i) To investigate the effect of nano calcium carbonate to the mechanical properties of epoxy composite reinforced recycle rubber.

(ii) To compared with each other specimen to see which weight percentage of nano $CaCO_3$ produces better mechanical properties.

1.4 Scopes of study

The scopes of this study mainly focusing are

(a) Mechanical properties of nano calcium carbonate to the mechanical properties of epoxy composite reinforced recycle ruber.

(b) Effects of vary wt% nano $CacO_3$ to mechanical properties of epoxy composite reinforced recycle rubber.

1.5 Research Methodology

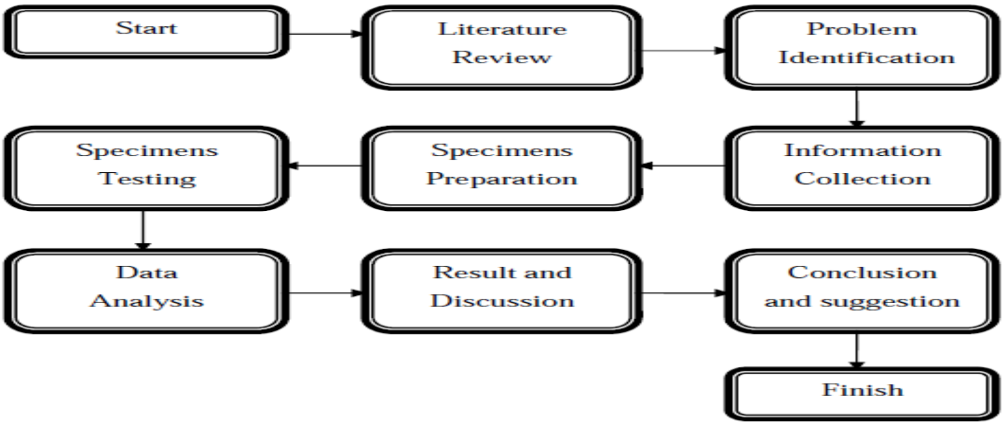


Figure 1.1: The Methodology Structure

In material selection process, it is establishing the link between material and function. It is important to start with the full menu materials in mind and failure to do so many mean a missed opportunity according by Michael (2005, p. 80). Figure 1.1 shows the project methodology which used in this study. The objectives and scopes of the project were defined at the beginning of the project. Literature review was done to understand the nature of the project and also involve the experiment and the scope study. The objective of the project was defined by the experiment.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Nanotechnology, shortened to "nanotech", is the study of the controlling of matter on an atomic and molecular scale. Generally nanotechnology deals with structures sized between 1 to 100 nanometer in at least one dimension, and involves developing materials or devices within that size (N. Taniguchi, 1974)

Nanotechnology is very diverse, ranging from extensions of conventional device physics to completely new approaches based upon molecular self-assembly, from developing new materials with dimensions on the nanoscale to investigating whether we can directly control matter on the atomic scale (Kahn and Jennifer, 2006)

There has been much debate on the future implications of nanotechnology. Nanotechnology may be able to create many new materials and devices with a vast range of applications, such as in medicine, electronics, biomaterials and energy production. On the other hand, nanotechnology raises many of the same issues as with any introduction of new technology, including concerns about the toxicity and environmental impact of nanomaterials, (Christina, *et al.*, 2007) and their potential effects on global economics, as well as speculation about various doomsday scenarios. These concerns have led to a debate among advocacy groups and governments on whether special regulation of nanotechnology is warranted.

PCC stands for Precipitated Calcium Carbonate also known as purified, refined or synthetic calcium carbonate. It has the same chemical formula as other types of calcium carbonate, such as limestone, marble and chalk: $CaCO_3$. The calcium, carbon and oxygen atoms can arrange themselves in three different ways, to form three different calcium carbonate minerals. The most common arrangement for both precipitated and ground calcium carbonates is the hexagonal form known as calcite. A number of different calcite crystal forms are possible: scalenohedral, rhombohedral and prismatic. Less common is aragonite, which has a discrete or clustered needle orthorhombic crystal structure. Rare and generally unstable is the vaterite calcium carbonate mineral. (http://www.specialtyminerals.com/our-minerals/what-is-pcc/ - online on 20 August 2010)

2.1.1 Calcium Carbonate

Calcium carbonate is a chemical compound with the chemical formula $CaCO_3$. It is a common substance found in rock in all parts of the world, and is the main component of shells of marine organisms, snails, pearls, and eggshells. Calcium carbonate is the active ingredient in agricultural lime, and is usually the principal cause of hard water. It is commonly used medicinally as a calcium supplement or as an antacid, but excessive consumption can be hazardous (Patnaik and Pradyot, 2003)

2.1.2 Chemical Properties of Calcium Carbonate

Calcium carbonate shares the typical properties of other carbonates. Notably:

- it reacts with strong acids, releasing carbon dioxide:
  $CaCO_{3(s)} + 2\ HCl_{(aq)} \rightarrow CaCl_{2(aq)} + CO_{2(g)} + H_2O_{(l)}$
- it releases carbon dioxide on heating (to above 840 °C in the case of $CaCO_3$), to form calcium oxide, commonly called quicklime, with reaction enthalpy 178 kJ / mole:
  $CaCO_3 \rightarrow CaO + CO_2$

Calcium carbonate will react with water that is saturated with carbon dioxide to form the soluble calcium bicarbonate.

$CaCO_3 + CO_2 + H_2O \rightarrow Ca\ (HCO_3)_2$

This reaction is important in the erosion of carbonate rocks, forming caverns, and leads to hard water in many regions (Solvay, 2007).

2.1.3 Preparation for Calcium Carbonate

The vast majority of calcium carbonate used in industry is extracted by mining or quarrying. Pure calcium carbonate (e.g. for food or pharmaceutical use), can be produced from a pure quarried source (usually marble).

Alternatively, calcium carbonate is prepared by calcining crude calcium oxide. Water is added to give calcium hydroxide, and carbon dioxide is passed through this solution to precipitate the desired calcium carbonate, referred to in the industry as precipitated calcium carbonate (PCC) (Solvay, 2007)

$$CaCO_3 \rightarrow CaO + CO_2$$

$$CaO + H_2O \rightarrow Ca(OH)_2$$

2.2 Nano Precipitated Calcium Carbonate

While the field of nano particle research has exploded in recent years, nano PCCs have been in commercial use for much longer. Specialty Minerals Inc. (SMI) has manufactured nano PCCs for more than 25 years. They have been widely used to make automotive and construction sealants and PVC window profiles. SMI's ultrafine PCCs range in particle size from 0.06 microns or 60 nanometers to 0.15 microns or 150 nanometers in median particle size. This is an order of magnitude smaller than the so-called ultrafine ground calcium carbonates, which are typically 0.7 microns. SMI's PCC tightly controlled precipitation.

2.2.1 Nano PCCs for Sealant Rheology and Reinforcement

With these extremely small particles, true thixotropic structure can be built in a sealant or other moderately to highly filled product in which control of viscosity, sag, slump and other rheological properties is needed. These ultrafine PCC particles also act as a semi-reinforcing filler, for strong physical performance. PVC plastisols, urethanes, silicones, polysulfides, and silylated polyethers are some of the types of high performance, long-lived automotive and construction sealants that use nano PCCs.

2.2.2 Nano PCCs for Rigid PVCs

In rigid polyvinyl chloride, nano PCCs can act as impact modifiers, providing the impact strength, even at very low temperatures, needed for PVC window profiles (which are used to form the frame of vinyl windows). When formulating with a nano PCC, replacing a larger sized ground calcium carbonate (GCC), the amount of expensive acrylic or chlorinated polyethylene (CPE) impact modifier used can be substantially reduced, saving money. Nano PCCs also give the highest gloss and best surface finish to PVC window profile extrusions.

2.2.3 Other Uses for Nano PCCs

Nano PCCs are also used in lithographic or offset inks. In highly filled litho inks, they can serve as the main rheological additive and cost-reducing filler. In lightly filled offset inks, they can extend other more expensive thickeners, as well as replace oils and varnishes. Nano PCCs thicken PVC plastisol silk screen inks. Gravure inks need very low abrasion fillers. Small particle-sized PCC is excellent here. Rheology and filling are also the reasons to use ultrafine PCCs in epoxy and other adhesives, as well as in unsaturated polyester gel coats.

Other uses include:

- i. High strength reinforcement of rubber
- ii. Detackification and mold release for thin gauge surgical and other medical gloves
- iii. Nucleating agent in emulsion polymerization
- iv. Carrier for catalysts, peroxides, fragrances
- v. Powder flow control additive for acrylic modifiers and other sticky products.

The uncoated nano PCC products can be used for fortifying beverages such as cow's milk and soy milk. By using a calcium carbonate with a very small particle size, the calcium fortifier stays suspended much longer, making a better tasting and appearing product.

2.3 Preparation for Precipitated Calcium Carbonate

Almost all PCC is made by direct carbonation of hydrated lime, known as the milk of lime process. The milk of lime process is simple in concept:

- i. Mine high purity calcium carbonate rock.
- ii. Crush the rocks to the particle size needed for processing – small stones or powder.
- iii. Separate some of the impurities from the crushed rock.
- iv. Calcine (heat) in a kiln to 1850° F, which takes the calcium carbonate apart, forming lime (CaO) and carbon dioxide gas (CO_2). The carbon dioxide can be captured for reuse.

  $\mathbf{CaCO_3 + Heat \rightarrow CaO + CO_2 \uparrow}$
- v. Add the lime to water to form calcium hydroxide (hydrated lime or slake).

  $\mathbf{CaO + H_2O \rightarrow Ca(OH)_2}$
- vi. Separate out additional impurities from the slaked lime.

vii. Combine the captured carbon dioxide with the slaked lime. Calcium carbonate reforms, and since it is insoluble in water, precipitates out.

$$\mathbf{Ca(OH)_2 + CO_2 \rightarrow CaCO_3 \downarrow + H_2O}$$

viii. Separate additional impurities and grit from the PCC slurry.

ix. If the PCC is to be used in a paper mill or shipped to a latex paint plant, the lower solids slurry may be used as is, or processed to bring up the solids level, then tested before transfer or shipment.

x. If the PCC is to be used as a dry product, the slurry is dewatered, dried, milled, packaged and tested.

While the process is simple on a laboratory scale, making precipitated calcium carbonates commercially on a large scale requires a great deal of process control and process technology to assure the right size, uniformity, shape, surface area and surface chemistry. This body of PCC technology developed by Specialty Minerals Research is what makes SMI PCCs outstanding in quality and consistency. (Freeman, *et al.*, 2002)

2.4 Manufacturing Process for Nano Precipitated Calcium Carbonate

Precipitated calcium carbonate is produced using the most economic process existing today. Limestone is converted into calcium oxide and carbon dioxide by means of calcination at temperatures in excess of 900°C. To ensure a high level of purity, the calcination process is carried out using natural gas. After the calcined lime has been slaked with water, the resulting milk of lime is purified and carbonated with the carbon dioxide obtained from the calcination process (See reactions bellow)

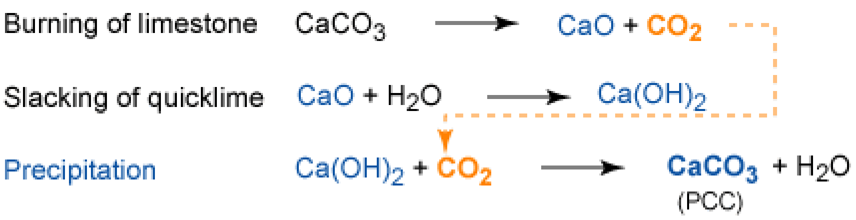


Figure 2.1: Reaction of Calcination Process

Following total carbonation, a suspension of CaCO3 results. A cake comprising 40% - 60% solid matter (depending on particle diameter) is then obtained by filtration. This filter cake is then dried and subsequently deagglomerated in grinders. Ultrafine PCC grades are reacted with fatty acids prior to filtration i.e. when still in the suspension stage (Freeman, *et al.*, 2002).

The fineness of the grain, as well as the crystal form (aragonite, calcite), is controlled by temperature, concentration of reactants and time.

Depending on the chemical composition of the milk of lime used and on the purifying stages during production, both technical as well as foodstuff and pharmaceutical grades can be produced (Freeman, *et al.*, 2002).

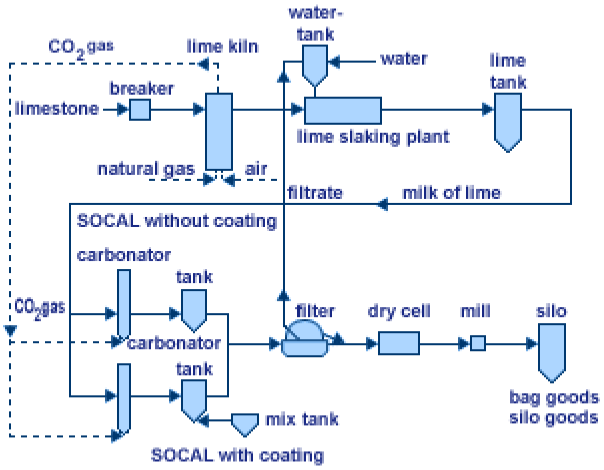


Figure 2.2: Manufacturing Process for Nano Precipitated Calcium Carbonate

2.5 Filler

Fillers are particles added to material (plastics, Composite material, Concrete) to lower the consumption of more expensive binder material or to better some properties of the mixtured material. Worldwide more than 50 million tons of fillers with a total sum of ca. 25 billion Euro for different application areas, such as paper, plastics, rubber, paints and adhesives, are consumed annually. As such, fillers, produced by more than 700 companies, rank among the world's major raw materials and are contained in a variety of goods for daily consumer needs (http://en.wikipedia.org/wiki/Filler_(materials) – online on – 28 August 2010)

Formerly, fillers were used predominantly to cheapen end products. Today, it has been proven that fillers are also able to enhance technical properties of the products. As a result, a number of optimized types of fillers, nano-fillers or surface treated goods have been developed. Among the 20 most important fillers, Calcium carbonate holds the largest market volume and is mainly used in the paper industry and increasingly in the plastics sector. While formerly ground calcium carbonate was used, today precipitated calcium carbonate which is manufactured with the natural mineral is increasingly applied to enhance technical properties of the end product.

2.6 Acrylonitrile-Butadiene Rubber (NBR)

Nitrile Rubber (NBR) is commonly considered the workhorse of the industrial and automotive rubber products industries. NBR is actually a complex family of unsaturated copolymers of acrylonitrile and butadiene. By selecting an elastomer with the appropriate acrylonitrile content in balance with other properties, the rubber compounder can use NBR in a wide variety of application areas requiring oil, fuel, and chemical resistance. In the automotive area, NBR is used in fuel and oil handling hose, seals and grommets, and water handling applications. With a temperature range of –40C to +125C, NBR materials can withstand all but the most severe automotive applications. On the industrial side NBR finds uses in roll covers, hydraulic hoses, conveyor belting, graphic arts, oil field packers, and seals for all kinds of plumbing and appliance applications. Worldwide consumption of NBR is expected to reach 368,000 metric tons annually by the year 2005.

Like most unsaturated thermoset elastomers, NBR requires formulating with added ingredients, and further processing to make useful articles. Additional ingredients typically include reinforcement fillers, plasticizers, protectants, and vulcanization packages. Processing includes mixing, pre-forming to required shape, and application to substrates, extrusion, and vulcanization to make the finished rubber article. Mixing and processing are typically performed on open mills, internal mixers, extruders, and calenders. Finished products are found in the marketplace as injection or transfer molded products (seals and grommets), extruded hose or tubing, calendered sheet goods (floor mats and industrial belting), or various sponge articles.

2.6.1 Chemistry and Manufacturing Process of NBR

NBR is produced in an emulsion polymerization system. The water, emulsifier/soap, monomers (butadiene and acrylonitrile), radical generating activator, and other ingredients are introduced into the polymerization vessels. The emulsion process yields a polymer latex that is coagulated using various materials (e.g. calcium chloride, aluminum sulfate) to form crumb rubber that is dried and compressed into bales. Some specialty products are packaged in the crumb form. Most NBR manufacturers make at least 20 conventional elastomer variations, with one global manufacturer now offering more than 100 grades from which to choose. NBR producers vary polymerization temperatures to make "hot" and "cold" polymers. Acrylonitrile (ACN) and butadiene (BD) ratios are varied for specific oil and fuel resistance and low temperature requirements. Specialty NBR polymers which contain a third monomer (e.g. divinyl benzene, methacrylic acid) are also offered [3, 4]. Some NBR elastomers are hydrogenated to reduce the chemical reactivity of the polymer backbone, significantly improving heat resistance (see HNBR product summary). Each modification contributes uniquely different properties. Figure 2 shows the typical NBR manufacturing process.

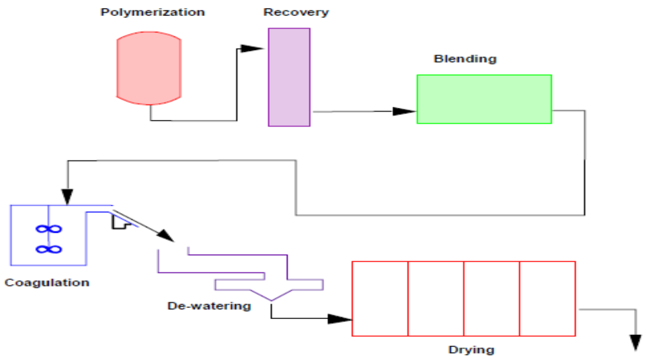
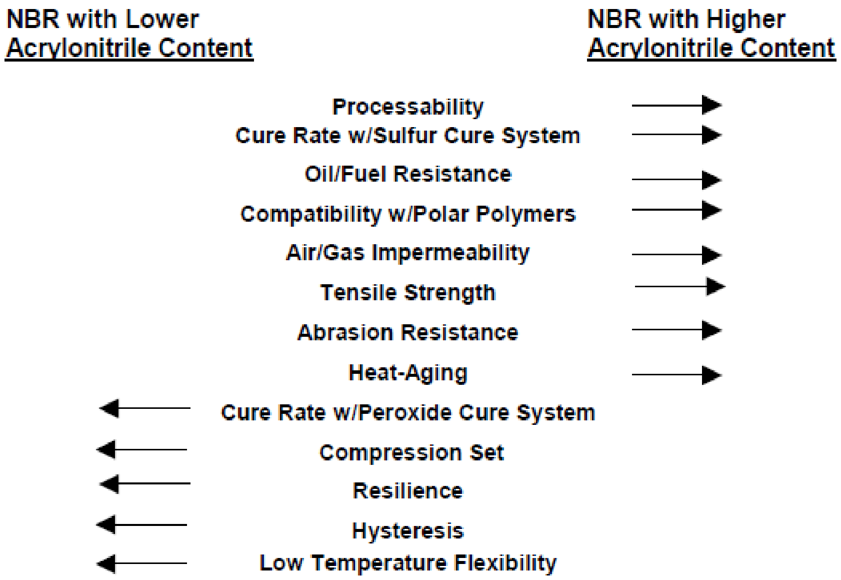


Figure 2.3: NBR manufacturing Process

2.6.2 Acrylonitrile (ACN) Content

The ACN content is one of two primary criteria defining each specific NBR grade. The ACN level, by reason of polarity, determines several basic properties, such as oil and solvent resistance, low-temperature flexibility/glass transition temperature, and abrasion resistance. Higher ACN content provides improved solvent, oil and abrasion resistance, along with higher glass transition temperature. Table 1 below summarizes most of the common properties for conventional NBR polymers. The direction of the arrows signifies an increase/improvement in the values.

Table 2.1 : NBR Properties – Relationship to Acrylonitrile Content (*Ibid.*, 71.)



NBR with Lower Acrylonitrile Content		NBR with Higher Acrylonitrile Content
	Processability	→
	Cure Rate w/Sulfur Cure System	→
	Oil/Fuel Resistance	→
	Compatibility w/Polar Polymers	→
	Air/Gas Impermeability	→
	Tensile Strength	→
	Abrasion Resistance	→
	Heat-Aging	→
←	Cure Rate w/Peroxide Cure System	
←	Compression Set	
←	Resilience	
←	Hysteresis	
←	Low Temperature Flexibility	

2.6.3 General Types of NBR

2.6.3.1 Cold NBR

The current generation of cold NBR’s spans a wide variety of compositions. Acrylonitrile content ranges from 15% to 51%. Mooney values range from a very tough 110, to pourable liquids, with 20-25 as the lowest practical limit for solid material. They are made with a wide array of emulsifier systems, coagulants, stabilizers, molecular weight modifiers, and chemical compositions. Third monomers are added to the polymer backbone to provide advanced performance. Each variation provides a specific function.Cold polymers are polymerized at a temperature range of 5 to 15°C , (Semon,W.L.,1954) depending on the balance of linear-to-branched configuration desired. The lower polymerization temperatures yield more-linear polymer chains. Reactions are conducted in processes universally known as continuous, semi-continuous and batch polymerization. Figure below shows the chemical structure of NBR, indicating the three possible isomeric structures for the butadiene segments.

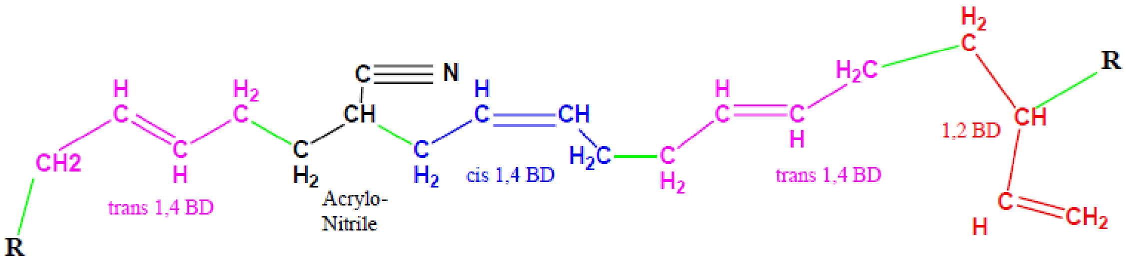


Figure 2.4: NBR Structure

2.6.3.2 Hot NBR

Hot NBR polymers are polymerized at the temperature range of 30 to 40°C[9]. This process yields highly branched polymers. Branching supports good tack and a strong bond in adhesive applications. The physically entangled structure of this kind of polymer also provides a significant improvement in hot tear strength compared with a cold-polymerized counterpart. The hot polymers' natural resistance to flow makes them excellent candidates for compression molding and sponge. Other applications are thin-walled or complex extrusions where shape retention is important (Horvath, J.W, 1990)

2.6.3.3 Crosslinked Hot NBR

Crosslinked hot NBR’s are branched polymers that are further cross-linked by the addition of a di-functional monomer. These products are typically used in molded parts to provide sufficient molding forces, or back pressure, to eliminate trapped air. Another use is to provide increased dimensional stability or shape retention for extruded goods and calendered goods. This leads to more efficient extruding and vulcanization of intricate shaped parts as well as improved release from calender rolls. These NBR’s also add dimensional stability, impact resistance, and flexibility for PVC modification. (Horvath, J.W, 1990)

2.6.3.4 Carboxylated Nitrile (XNBR)

Addition of carboxylic acid groups to the NBR polymer's backbone significantly alters processing and cured properties (Horvath, J.W, 1990). The result is a polymer matrix with significantly increased strength, measured by improved tensile, tear, modulus and abrasion resistance. The negative effects include reduction in compression set, water resistance, resilience and some low-temperature properties

2.6.3.5 Bound Antioxidant NBR

Nitrile rubbers are available with an antioxidant polymerized into the polymer chain (Kline, R, 1974). The purpose is to provide additional protection for the NBR during prolonged fluid service or in cyclic fluid and air exposure. When compounding with highly reinforcing furnace carbon black the chemical reactivity between the polymer and the pigment can limit hot air aging capability. Abrasion resistance is improved when compared with conventional NBR, especially at elevated temperatures. They have also been found to exhibit excellent dynamic properties (Horvath, J.W, 1990)

2.7 Polybutadiene (BR)

2.7.1 Properties and Applications

Polybutadiene (BR) is the second largest volume synthetic rubber produced, next to styrene-butadiene rubber (SBR). Consumption was about 1,953,000 metric tons worldwide in 1999. The major use of BR is in tires with over 70% of the polymer produced going into treads and sidewalls. Cured BR imparts excellent abrasion resistance (good tread wear), and low rolling resistance (good fuel economy) due to its low glass transition temperature (T_g). The low T_g , typically <–90C, is a result of the low “vinyl” content of BR, which will be discussed below. Nitrile rubber is more resistant than natural rubber to oils and acids, but has inferior strength and flexibility. Nitrile gloves are nonetheless three times more puncture-resistant than rubber gloves (Crystal and Garry, 2008). However, low T_g also leads to poor wet traction properties, so BR is usually blended with other elastomers like natural rubber or SBR for tread compounds. BR also has a major application as an impact modifier for polystyrene and acrylonitrile-butadiene-styrene resin (ABS) with about 25% of the total volume going into these applications. Typically about 7% BR is added to the polymerization process to make these rubber-toughened resins (see picture below). Also, about 20,000 metric tons worldwide of "high cis" polybutadiene is used each year in golf ball cores due to its outstanding resiliency. This application is growing since the golf ball industry seems to be moving away from the traditional wound ball technology to the two-piece, solid core construction (Mackey and Jorgensen, 1999)

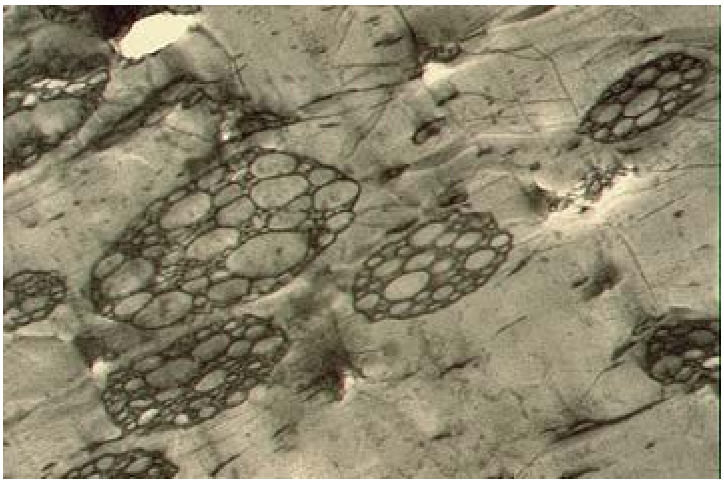


Figure 2.5: Electronmicrograph (5,600X) Rubber-toughened polystyrene resin with BR domains (shown in black)

2.7.2 Chemistry and Manufacturing Process

Polybutadiene is a homopolymer (only one monomer) of 1,3 butadiene, a monomer containing four carbon atoms, and six hydrogen atoms (C_4H_6). The four carbon atoms are in a straight chain containing two “double bonds” as follows:

CH_2=CH-CH=CH_2
1,3 butadiene

It is the double bonds that are the key to polymer formation. They are attacked by catalysts to maintain a repetitive chain growth process which continues until something is added to terminate the reaction at the desired molecular weight. Molecular weight can become quite high. For a typical BR, molecular weight (M_n = number average) is usually >100,000 grams per mole. This represents a chain that contains over 2,000 butadiene units. Most BRs are made by a solution process, using either a transition metal (Nd, Ni, or Co) complex or an alkyl metal, like butyllithium, as catalyst. Since the reaction is very exothermic, and can be explosive, particularly with alkyllithium catalysts, the reaction is normally carried out in solvents like hexane, cyclohexane, benzene or toluene. The solvents are used to reduce the rate of reaction, control the heat generated by the polymerization and to lower the viscosity of the polymer solution in the reactor. A typical BR polymerization would be run at about 20% monomer and 80% solvent. The polymerization can either be a batch process or a continuous process. In batch mode, monomer, solvent and catalyst are charged to the reactor, heated to initiate the process, and then allowed to continue to completion. The polymer solution is then transferred to another vessel or process unit to remove the solvent. In continuous mode, monomer, solvent and catalyst are continuously fed into the bottom of the first of a series of reactors at a temperature suitable for polymerization. The polymerization progresses as the solution flows through the reactors and polymer solution is taken off at the top of the last reactor without stopping the process. The continuous process is the most economical. In both processes, the finished product is usually in the form of bales which weigh from 50 to 75 pounds each (Hoffman, 1964)

2.7.3 High Cis Polybutadiene

The alkyllithium and transition metal catalysts make very different products. The transition metal, or so called Ziegler catalysts produce very "stereoregular" BRs with one type having the main polymer chain on the same side of the carbon-carbon double bond contained in the polybutadiene backbone. This is called the cis configuration.

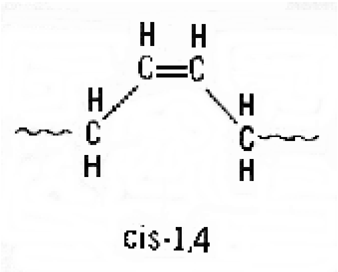


Figure 2.6: High Cis Polybutadiene

High cis BR will usually have cis content >95% which gives rise to better "green strength" and increased cut growth resistance in the cured product. Green strength, which is the strength of the uncured rubber compound, is important for the tire building process and cut growth resistance is necessary for tire performance. Cut growth resistance is the resistance to the propagation of a tear or crack during a dynamic operation like the flexing of a tire in use. High cis BR also shows lower T_g compared to alkyllithium-based BR because it has almost no vinyl structure. As mentioned earlier, vinyl tends to increase the T_g of the polymer. The low vinyl content and low T_g makes high cis BR ideal for golf ball cores. Golf ball cores are cured with peroxides, which tend to "over cure" the vinyl units making a very hard and slow golf ball. The neodymium catalyst system produces the highest cis content of about 99% and also makes the most linear chain structure (no branching) producing a polymer with the best tensile and hysteresis (low heat build-up) properties of all the high cis types. The cobalt system produces a highly branched BR with a low solution viscosity that makes a good polystyrene and ABS modifier. The nickel catalyst makes BR with an intermediate level of branching (Kline, 1974)

2.7.4 Lithium-based Polybutadiene

The alkyllithium or "anionic" catalyst system produces a polymer with about 40% cis, 50% trans and 10% vinyl when no special polar modifiers are used in the process. The alkyllithium process is probably the most versatile, because the growing chain end contains a "living" anion (negative charge) which can be further reacted with coupling agents or functional groups to make a variety of modified BRs. It also produces gel-free BR making it ideal for plastics modification. Vinyl increases the T_g of the polybutadiene by creating a stiffer chain structure. Vinyl also tends to crosslink or "cure" under high heat conditions so the high vinyl polymers are less thermally stable than low vinyl. Note above, that in vinyl units the double bonds are pendent to the main chain, giving rise to the special properties of high vinyl polymers. Vinyl units can be increased in lithium-based anionic polymerization through the use of polar modifiers, which are usually nitrogen or oxygen-containing compounds. The modifiers direct the attack of the propagating anion on the "living" chain end to give a 1,2 addition to the butadiene monomer.

$CH_3 – CH{=}{=}CH – CH_2 – (CH_2 – CH{=}{=}CH – CH_2)_n – CH_2 – CH{=}{=}CH –$
$CH_2\ Li \oplus$

Growing "living" anion (negative charge) on end of live polybutadiene chain with Lithium counterion (positive charge) (Kline, 1974)

2.7.5 High Trans Polybutadiene

High trans BR is a crystalline plastic material similar to high trans polyisoprene or balata, which was used in golf ball covers. Note below, that in the trans configuration the main polymer chain is on opposite sides of the internal carbon-carbon double bond. Trans BR has a melting point of about 80C. It is made with transition metal catalysts similar to the high cis process (La, Nd, and Ni). These catalysts can make polymers with >90% trans again using the solution process. (Kline, 1974)

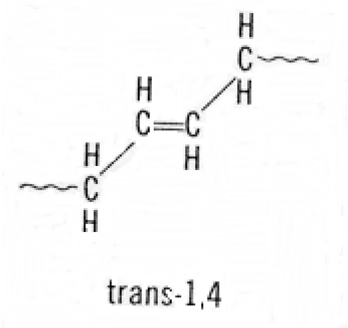


Figure 2.7: High Trans Polybutadiene

2.8 Styrene Butadiene Rubber (SBR)

SBR stands for Styrene – Butadiene Rubber that is a random copolymer from the aforesaid monomers. SBR is a synthetic rubber copolymer consisting of styrene and butadiene (Mallick, 1993) It has good abrasion resistance and good aging stability when protected by additives, and is widely used in car tires, where it is blended with natural rubber. It was originally developed prior to World War II in Germany, (http://www.wisegeek.com/what-is-styrene-butadiene-rubber.htm - online on 1 September 2010) There are two major types of SBR, Emulsion SBR (E-SBR) and Solution SBR (S-SBR), based on the different manufacturing process. The peculiar nature of the insertion of butadiene on the growing chain, ie the 1,4 and 1,2 additions, together with the two possible 1,4 addition isomers, cis and trans, suggests that it would be more appropriate to refer to SBR as a four-monomer copolymer. This remark acquires a particular meaning if we consider the physical and rheological characteristics of the finished polymer. The balance between the structural unit content of styrene, 1,4 and 1,2 butadiene along the chain is the most important parameter affecting the Tg of the material. Most interestingly, the concentration of 1,4 trans units has a strong influence on the strain induced crystallization of the rubber, which means a reinforcing effect on the tensile ultimate properties: in this sense, natural rubber shows a strain induced crystallization behavior. Moreover, the relative concentration of 1,4 and 1,2 units may influence the thermal stability of the polymer. The oxidative degradation of the rubber starts from the addition of oxygen on a double bond: if the double bond id part of the main chain, as in the case of 1,4 units, the reaction will lead to a chain scission.

2.9 Polymer

Nowadays, the polymer industry has grown to be larger than the metals industries, as polymers have already had a range of applications in nowadays industry. These included adhesive, coatings, foams and even to packaging materials to textile and industrial fibers industry. Polymer is a large molecule (macromolecule) composed of repeating structural units typically connected by covalent chemical bonds. Plastic is the most synonyms with this term and this term actually refers to a large class of natural and synthetic materials with a variety of properties. Polymers have become an essential and ubiquitous role in everyday life due to the extraordinary range of properties accessible in polymeric materials, from plastics and elastomers on the one hand to natural biopolymers such as DNA and proteins that are essential for life on the other. A simple example is polyethylene, whose repeating unit is based on ethylene (IUPAC name *ethene*) monomer. Most commonly, as in this example, the continuously linked backbone of a polymer used for the preparation of plastics consists mainly of carbon atoms.

However, other structures do exist; for example, elements such as silicon form familiar materials such as silicones, examples being silly putty and waterproof plumbing sealant. The backbone of DNA is in fact based on a phosphodiester bond, and repeating units of polysaccharides (e.g. cellulose) are joined together by glycosidic bonds via oxygen atoms. Natural polymeric materials such as shellac, amber, and natural rubber have been in use for centuries. The list of syntheticpolymers includes synthetic rubber, Bakelite, neoprene, nylon, PVC, polystyrene, polyethylene, polypropylene, polyacrylonitrile, PVB,silicone, and many more.(Wikipedia 2009)

2.10 Epoxy

Epoxy or polyepoxide is a thermosetting polymer formed from reaction of an epoxide "resin" with polyamine "hardener". Epoxy has a wide range of applications, including fiber-reinforced plastic materials and general purpose adhesives.

2.11 Chemistry of Epoxy

Epoxy is a copolymer; that is, it is formed from two different chemicals. These are referred to as the "resin" and the "hardener". The resin consists of monomers or short chain polymers with an epoxide group at either end. Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A, though the latter may be replaced by similar chemicals. The hardener consists of polyamine monomers, for example Triethylenetetramine (TETA). When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond. Each NH group can react with an epoxide group, so that the resulting polymer is heavily crosslinked, and is thus rigid and strong.

The process of polymerization is called "curing", and can be controlled through temperature, choice of resin and hardener compounds, and the ratio of said compounds; the process can take minutes to hours. Some formulations benefit from heating during the cure period, whereas others simply require time, and ambient temperatures.

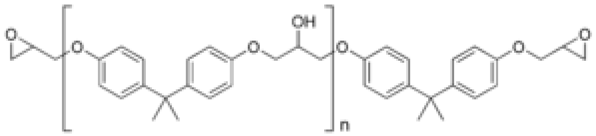


Figure 2.8 : Structure of unmodified epoxy prepolymer resin

CHAPTER 3

METHODOLOGY

3.1 Introduction

Methodology means to describe on the overall processes and component that involved in the whole scope of the study to achieve the objective of the study. This includes raw material characterization, sample preparation, mechanical testing, and analysis of the result obtained. The purpose of having a methodology is some of system of broad rules from which specific method and procedure to make sure this report complete.

3.2 Flow Chart of Methodology

This project is start from preparing of sample by adding the epoxy resin with recycle rubber with ratio 1 : 0.4. The material were stir using mechanical stirrer at speed 500rpm until it mixed. After that nano $CaCO_3$ powder will added with 5 different weight percent (0%, 2%, 4%, 6%, and 8%).. The nano-$CaCO_3$ powder was added and mixed using the mechanical stirrer mill at speed 500 rpm for another 5 min. Then the hardener were added in the same manner, at speed 500 rpm for 5 min. After the mixture is prepared, this mixture will poured into the mould and then wait about 5 hour until it cured. Then the sample will cured inside the furnace and be heated at 140 °C for 1 hour . The resultant nano $CaCO_3$ + epoxy composite reinforced recycle rubber of about 3 mm in thickness. After that, the mechanical properties of samples will analysis using Shimadzu Universal Tensile Machine AG-1, Scanning Electron Microscope (SEM), impact test and microhardness test.

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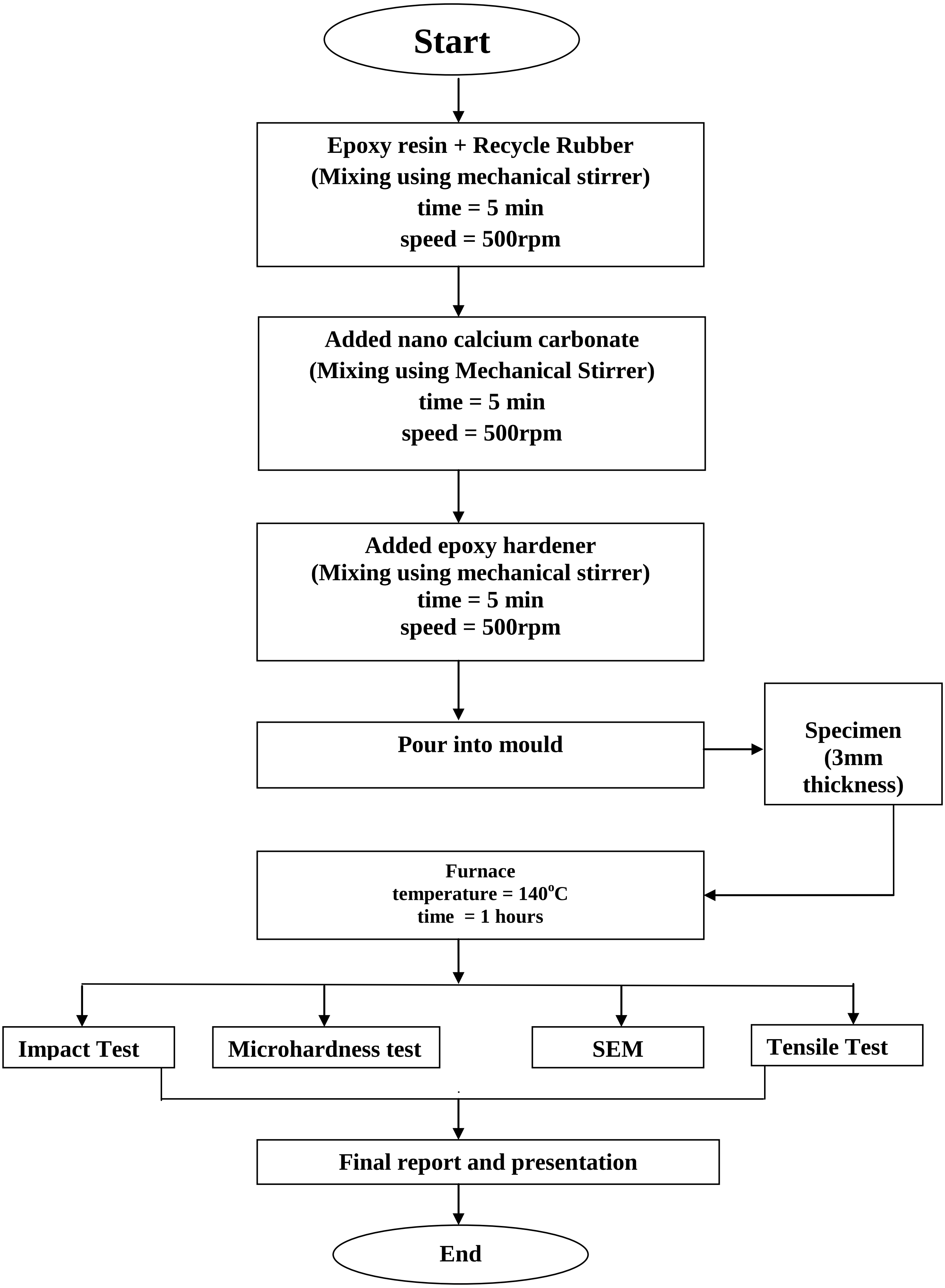


Figure 3.1 : Process Flow Chart

3.3 Sample preparation

The materials used in this project are nano calcium carbonate powder (nano $CaCO_3$), recycle rubber (BR and SBR), epoxy resin and epoxy hardener. These materials are mixed using vary nano $CaCO_3$ powder particle size and percent content. The nano-size calcium carbonate (nano-$CaCO_3$) used in this study, 20nm, was supplied by Maju Tek.

The compounding formulations are summarized in Table 1. The mixing ratio of the epoxy, hardener, recycle rubber and nano CaCO3 powder was kept constant for all experiments as 1:0.7:0.4 by weight, respectively. In this study five different weight percent (0%, 2%, 4%, 6%, and 8 wt%) of nano CaCO3 powder will be vary too.

Table 3.1 : Compounding formulation

Weight % of Nano $CaCO_3$	**Weight (gram)**			
	Epoxy Resin	**Epoxy Hardener**	**Recycle Rubber**	**Nano $CaCO_3$ Powder**
0	**100**	**70**	**40**	**0**
2	**100**	**70**	**40**	**4.2**
4	**100**	**70**	**40**	**8.4**
6	**100**	**70**	**40**	**12.6**
8	**100**	**70**	**40**	**16.8**

3.3.1 Mechanical Stirring

The tecycle rubber rubber, epoxy resin, epoxy hardener and nano CaCO3 were performed using a mechanical stirring and heated at temperature 60 °C for 5 min. The recycle rubber and epoxy resin was added and mixed using the mechanical stirrer mill at speed 500 rpm for 5 min. Then nano-$CaCO_3$ powder were added in the same manner, at speed 500rpm for 5 min. Beside that the mechanical stirring also been applied to give the shear force to make this mixture fully homogenized.

Figure 3.2 : Mechanical Stirrer Apparatus

3.3.2 Furnace

A furnace can heat materials, typically metals, to very high temperatures and carry out processes such as brazing, sintering and heat treatment with high consistency and low contamination.

In a vacuum furnace the product in the furnace is surrounded by a vacuum. The absence of air or other gases prevents heat transfer with the product through convection and removes a source of contamination. Some of the benefits of a vacuum furnace are:

- Uniform temperatures in the range 2000–2800°F (1100–1500°C)
- Temperature can be controlled within a small area
- Low contamination of the product by carbon, oxygen and other gases
- Quick cooling (quenching) of product.
- The process can be computer controlled to ensure metallurgical repeatability.

Heating metals to high temperatures normally causes rapid oxidation, which is undesirable. A vacuum furnace removes the oxygen and prevents this from happening

3.3.3 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is a powerful analytical tool which directly measures the stability and unfolding of a protein, lipid, or nucleic acid. In DSC, the biomolecule is heated at a constant rate and there is a detectable heat change associated with thermal denaturation. DSC measures the temperatures and heat flows associated with transitions in materials as a function of temperature or time in a controlled atmosphere. This technique provides quantitative and qualitative information about physical and chemical changes that involve endothermic or exothermic processes, or changes in heat capacity.

In a single DSC experiment, can determine:

- Transition midpoint - T_m
- Enthalpy (ΔH) and heat capacity change (ΔCp) associated with unfolding

A biomolecule in aqueous solution is in equilibrium between the native (folded) conformation and its denatured (unfolded) conformation. The stability of the native state is based on the magnitude of the Gibbs free energy (ΔG) of the system and the thermodynamic relationships between enthalpy

(ΔH) and entropy (ΔS) changes. A positive ΔG indicates the native state is more stable than the denatured state – the more positive the ΔG, the greater the stability. For a protein to unfold, stabilizing forces need to be broken. Conformational entropy overcomes stabilizing forces allowing the protein to unfold at temperatures where entropy becomes dominant.

DSC measures ΔH of unfolding due to heat denaturation. The transition midpoint T_m is the temperature where 50% of the protein is in its native conformation and the other 50% is denatured. The higher the T_m, the more stable the molecule. During the same experiment DSC also measures the change in heat capacity (ΔCp) for denaturation. Heat capacity changes associated with protein unfolding are primarily due to changes in hydration of side chains that were buried in the native state, but become solvent exposed in the denatured state.

Many factors are responsible for the folding and stability of native biopolymers, including hydrophobic interactions, hydrogen bonding, conformational entropy, and the physical environment (pH, buffer, ionic strength, excipients, etc.)

Figure 3.3 : Differential Scanning Calorimetry

3.3.4 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope is enabling to examination of the surface morphology (Abdolldhi *et al,* 2009) and microstructure of the sample. The sample is the oxidised aluminium alloy and titanium alloy. The microstructure of oxidised aluminium alloy and titanium alloy was investigated to look the surface feature and characterize the oxide layer thickness of the sample.

SEM magnify is about 10x to 500Kx and the resolution is 1.5nm is very good to look morphology shape and size of the particles making up the sample. The SEM expands the resolution range by more than two orders of magnitude to approximately 4 nm in routine instruments, with ultimate values below 1 nm. The SEM typical operates at 2 to 50kV and the electromagnetic lenses are used to from a small diameter electron probe. A set of scan coils raster the electron probe over the specimen surface as an electron beam scans the inside of the CRT screen. Figure 3.5 is show the SEM machine.

Figure 3.4 : Scanning Electron Microscope (UTeM)

3.3.5 Tensile Test

In this project, tensile machine that used is Shimadzu Universal Tensile Machine AG-1 (100kN). First of all, machine test will be prepared based on manual of the machine. Then, the gage length marking of test specimen should be determined. This step must be done to determination of elongation must be accordance with product specification.

Figure 3.5 : Shimadzu Universal Tensile Machine AG-1

Next step, sample will be grip at testing machine. The grip will be lowered according with the specimen sizes. Machine speed must be set depend on sample condition before the machine started. At the same time, student must follow all instruction regarding tensile machine operation setting that shown by technician.

From the theory, when there occur strain on the specimen is roughly forces imposed, then to test the strength of the rubber, specimen can be calculate using tensile strength, □and tensile modulus, E formula.

(a) Tensile Strength, ε

$$\varepsilon \text{ (MPa)} = \frac{P \text{ (N)}}{A \text{ (mm}^2\text{)}}$$

(b) Tensile Modulus, E

$$E\ (\text{MPa}) = \frac{\text{Tensile Stress } (\sigma)}{\text{Tensile Strain } (\varepsilon)}$$

3.3.6 Impact Test

Impact testing is designed to measure the performance of an object under high-rate loading. It can be a method for evaluating the toughness and notch sensitivity of engineering materials. It is usually used to test the toughness of metals, but it also used for polymers, ceramics and composites. The notched test specimen is broken by the impact of a heavy pendulum or hammer, falling at a predetermined velocity through a fixed distance. The test measures the energy absorbed by the fractured specimen.

Generally, impact testing can be classified into two categories. There are low velocity and high velocity impact test. Low velocity impact test is generally done by using a drop-hammer or swinging pendulum involving a relatively large mass. While for the high velocity impact, it is using a gas gun or some other ballistic launcher which is mainly concern with small masses. For low velocity impact testing, there have two types of testing commonly used which are the Izod and Charpy test. They have been used to measure the impact performance of materials particularly with respect to brittle or ductile transition temperature and notch sensitivity. For Izod test, it is consists a rectangular or square cross section bar of specified dimensions is clamped at one end and struck towards the top of the test piece with a pendulum. For this project, it will focus on Charpy test only.

It was realized that the result of tensile test could not predict a fracture mode for example under some circumstances normally ductile materials fracture abruptly with very little plastic deformation. So, impact test were chosen to represent those most severe relative to the potential for fracture namely is deformation at a relatively low temperature, a high strain rate, and a triaxial stress rate. In addition, the notched test specimen is broken by the impact of a heavy pendulum or hammer, falling at a predetermined velocity through a fixed distance in impact testing.

3.3.7 Charpy Test

The Charpy impact test is a standardized high strain-rate test of standard notched specimen which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's toughness and acts as a tool to study temperature-dependent brittle-ductile transition. Materials are used to build load-bearing structures. It is important to know if the material will survive the conditions that the structure will see in service. Important factors that adversely affect the toughness of a structure include low-test temperatures, extra loading and high strain rates due to over pressurization or impacts and the effect of stress concentrations such as notches and cracks. These all tend to encourage fracture.

In certain extent, the complex interaction of these factors can be included in the design process by using fracture mechanics theory. The impact test is a method for evaluating the relative toughness of engineering materials. The Charpy impact test continues to be used nowadays to assess the notch sensitivity and impact toughness of engineering materials. It is usually used to test the toughness of metals. Similar tests can be used for polymers, ceramics and composites.

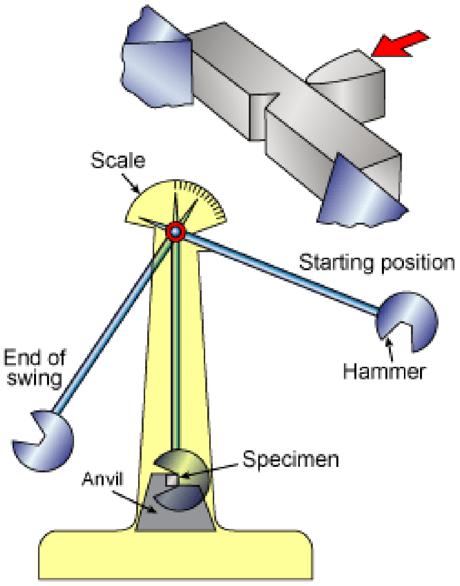


Figure 3.6 : Apparatus for Charpy impact testing

For the Charpy test, the specimen is in the shape of square cross section was machining to V- notch. The load that applied as an impact is blow from a cooked position at a fixed height, h. The specimen is positioned at the base as shown. Upon release, a knife edge mounted on the pendulum strikes and fractures the specimen at the notch, which acts as a point of stress concentration for this high velocity impact blow. Then, the pendulum will continues swing, until rising to a maximum height h’,
which is lower than its original height, h. (Ling Yih Lii, 2009)

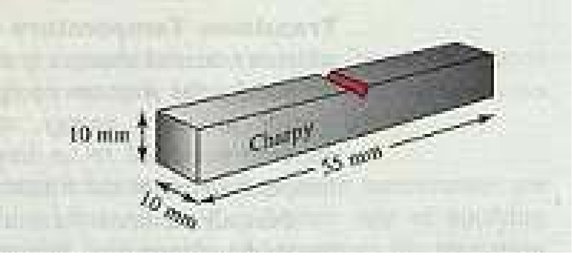
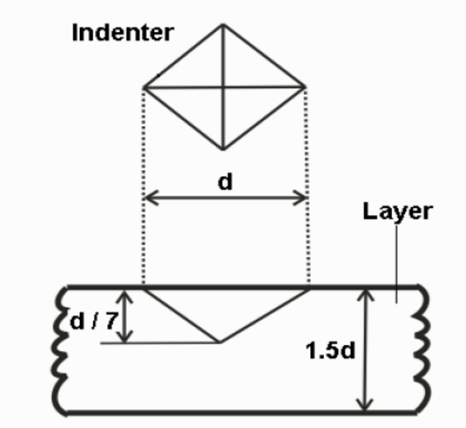


Figure 3.7 : The standard V-notched Charpy test specimen

The impact energy can be measured from the difference between h and h’. The test results may influence by the variables including specimen size and shape. The notch in the sample also affects the results of the impact test, thus it is necessary for the notch to be of a regular dimensions and geometry. The Charpy impact tests determine the fracture properties of materials which may be used for design purposed.

3.3.8 Hardness Test (Vickers Micro Hardness)

The hardness test was done on the specimen surface when oxidising process is completed. The Micro Vickers Hardness Test sample offers to indentation by an indenter which is a pyramid-shaped diamond under an applied load. The micro hardness is the ratio of load to the surface area of the pyramid-shaped permanent indentation. The figure 3.3 shows the shape of the tip of a Vickers Micro hardness indenter and the symbols used in subsequent equation. The testing method of Vickers Micro Hardness Test was followed the Standard Test Method for Microindentation Hardness of Material (ASTM E 384 – 99) to conduct the testing on coating or substrate material. The indentation depth in investigating layer or coating on a substrate material, the thickness of the layer has to be considered. It should have minimum thickness in respect to the diagonal length, which is proportional to the indentation depth. The minimum layer thickness should be approximately 10 times larger than the indentation depth. Base on the geometric shape of Vickers correlation between diagonal length, indentation depth and minimum layer thickness. Figure 3.3 show the indentation depth and equation of Vickers Micro hardness. Figure 3.4 is show the Vickers Micro hardness Test equipment.



d: Diagonal length

t : Minimum layer thickness ***(1.5d)***

h: Indentation depth ***(d/7)***

4d: Minimum distance to next print

2.5 d: Minimum distance to specimen edge

F: Force Kgf

$$HV = 0.1891\ F/d^2$$

Figure 3.8: Indentation depth and equation of Vickers micro hardness on coating or substrate.

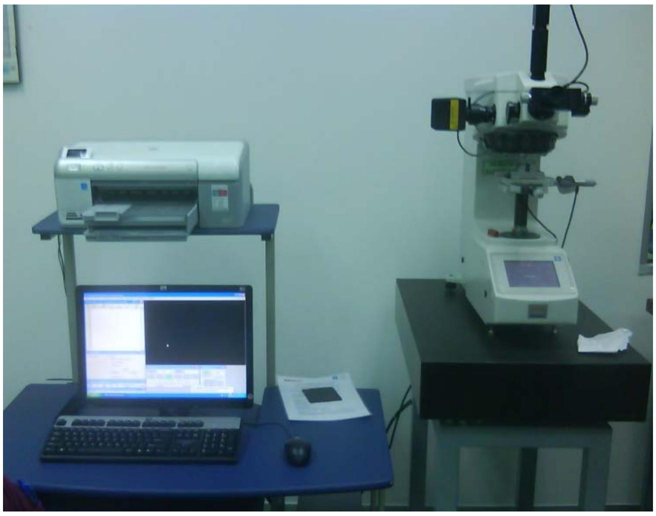


Figure 3.9: Vickers micro hardness Test (UTeM)

CHAPTER 4

RESULTS AND DISCUSSIONS

4.0 Introduction

In this chapter, all the data and results will be presented to see the effect of nano-$CaCO_3$ on mechanical properties of epoxy composite reinforced recycle rubber. In this chapter also will be discussed the reason why all the specimen behave the way it gave it mechanical properties and how does the nano-$CaCO_3$ change their morphology structure. There were three tests that conducted in order to determine the mechanical properties of the specimen. The tests that conducted were tensile tests, microhardness tests and Charpy tests. In this chapter also will be discussing the results and interpreted it.

4.1 Tensile Tests Results and Discussion

The tests were conducted on the recycle rubber and nano-$CaCO_3$ toughened epoxy composite with different percentage weight % of nano-$CaCO_3$. The tensile tests were conducted using Universal testing machine (UTM). The main objective for this test is to determine the maximum stress, load, displacement, Young Modulus that acting on this material. This test is also to study the behavior of the specimens under different axial load.

Figure below shows the result of tensile test which is to find the longitudinal young's modulus, maximum stress, maximum strain, maximum force and behavior of the specimen.

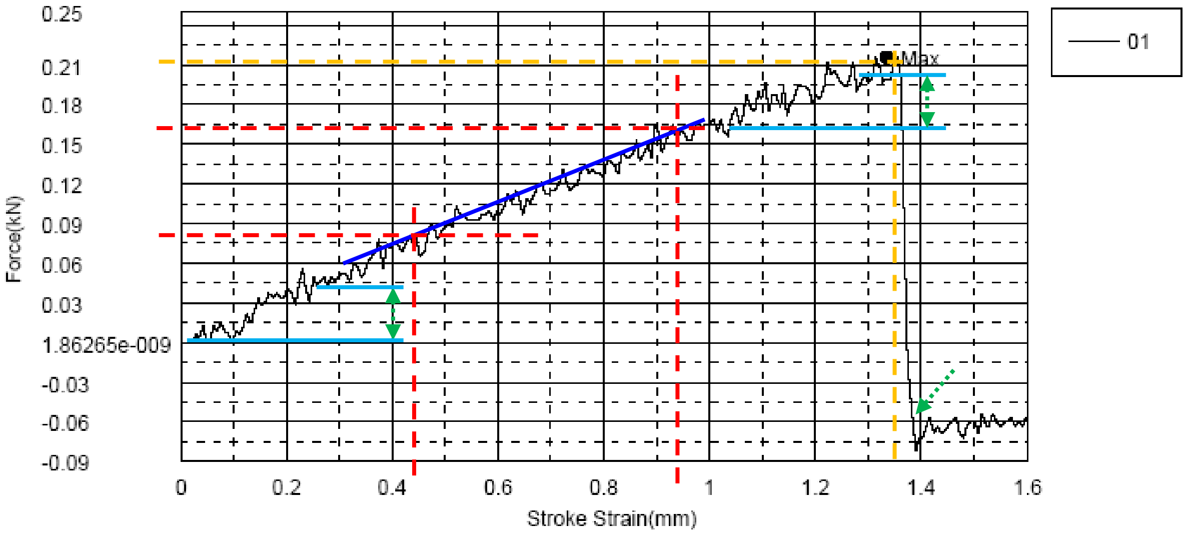


Figure 4.1: Result tensile test 0% of nano-$CaCO_3$

Stress, σ_1

$$\sigma_1 = \frac{F_1}{A_1}$$

$$= \frac{0.078 \text{ x } 10^3 \text{ N}}{3.81 \text{ x } 10^{-5} \text{ m}^2}$$

$$= 2.04 \text{ x } 10^6 \text{ N/m}^2 \qquad (4.1)$$

Stress, σ_2

$$\sigma_2 = \frac{F_2}{A_2}$$

$$= \frac{0.16 \text{ x } 10^3 \text{ N}}{3.81 \text{ x } 10^{-5} \text{ m}^2}$$

$$= 4.19 \text{ x } 10^6 \text{ N/m}^2 \qquad (4.2)$$

Strain, ε

$$\varepsilon = \frac{S_2 - S_1}{S_1}$$

$$= \frac{0.95 \text{ x } 10^{-3}\text{m} - 0.44 \text{ x } 10^{-3}\text{ m}}{0.44 \text{ x } 10^{-3}\text{ m}}$$

$$= 1.16 \tag{4.3}$$

Young Modulus, E

$$E = \frac{\sigma_2 - \sigma_1}{\varepsilon}$$

$$= \frac{4.19 \text{ x } 10^6 \text{ N/m}^2 - 2.04 \text{ x } 10^6 \text{ N/m}^2}{1.16}$$

$$= 1.853 \text{ MPa} \tag{4.4}$$

Stress maximum

$$\sigma_{\text{max}} = \frac{F_{\text{max}}}{A}$$

$$= \frac{0.214 \text{ x } 10^3 \text{ N}}{3.81 \text{ x } 10^{-5} \text{ m}^2}$$

$$= 5.617 \text{ x } 10^6 \text{ N/m}^2$$

$$= 5.6 \text{ MPa} \tag{4.5}$$

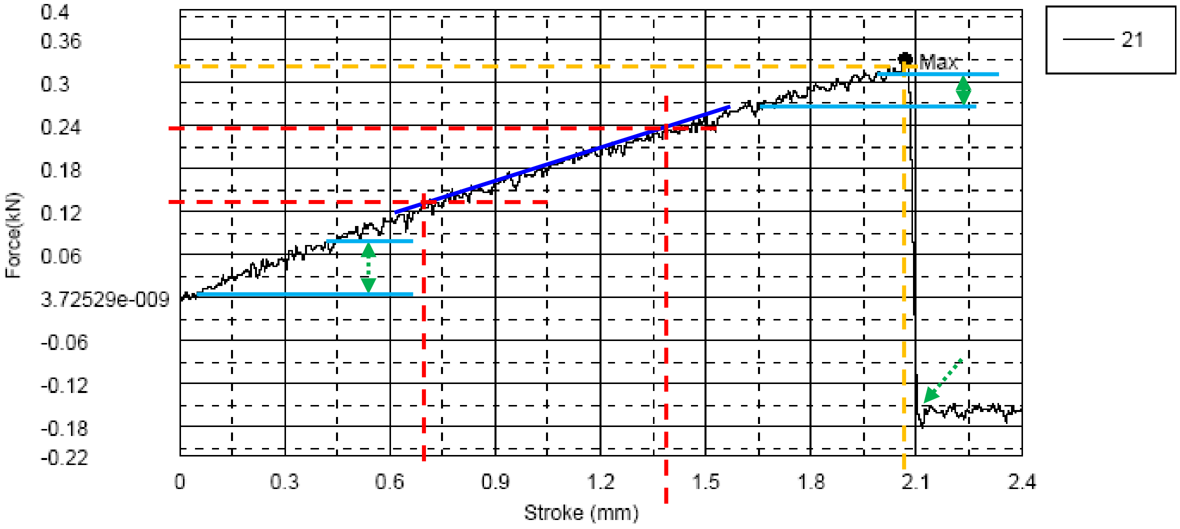


Figure 4.2: Result tensile test 2% of nano-$CaCO_3$

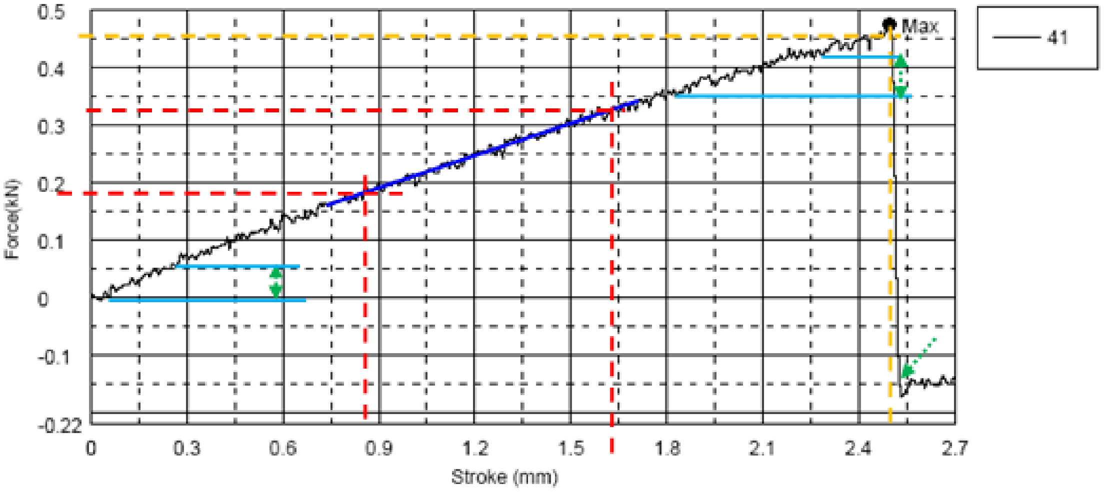


Figure 4.3 : Result tensile test 4% of nano-$CaCO_3$

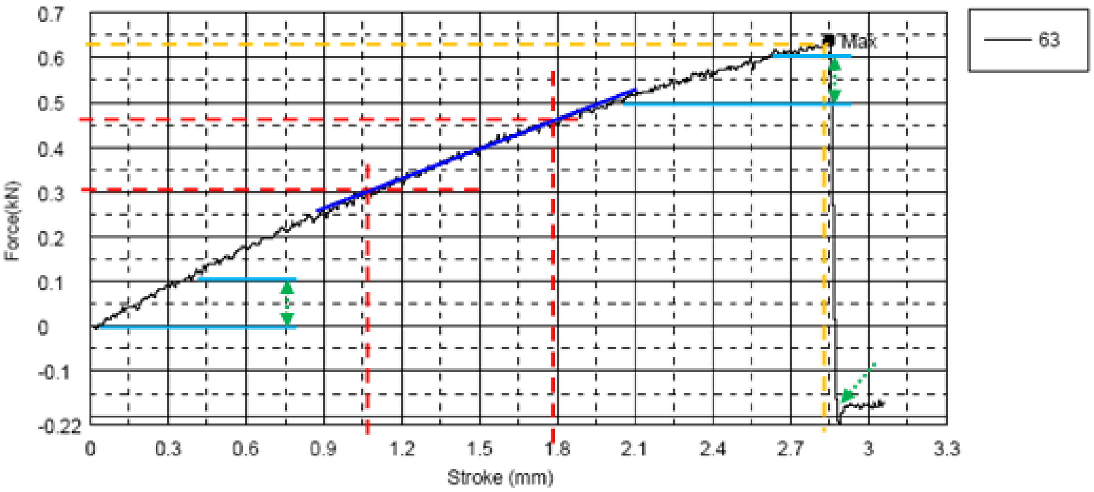


Figure 4.4 : Result tensile test 6% of nano-$CaCO_3$

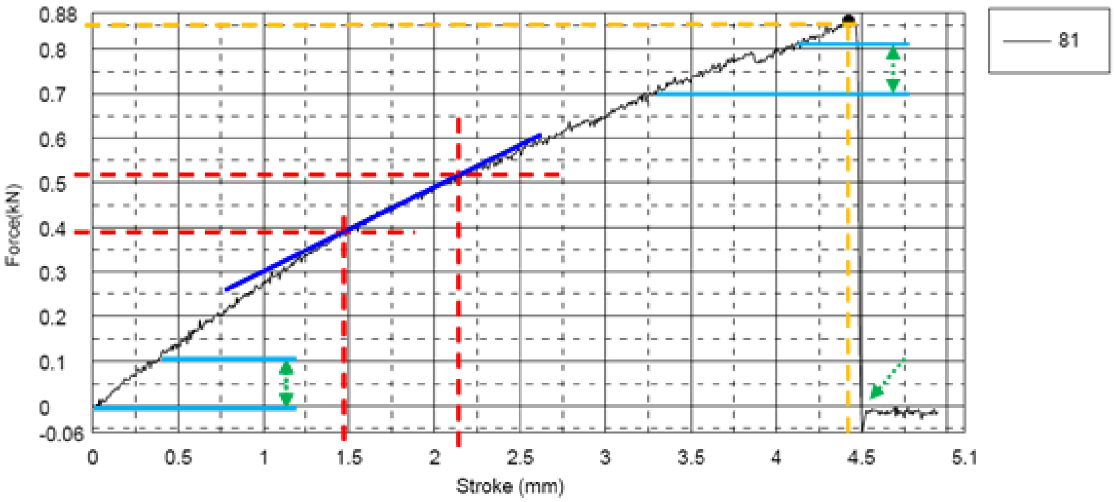


Figure 4.5: Result tensile test 6% of nano-$CaCO_3$

Table 4.1 : Tensile Test Result

% wt of nano-CaCO$_3$	F Max(kN)	ε Max(m)	σ Max(MPa)	E (MPa)
0	0.214	0.0013	5.6	1.853
2	0.33125	0.0021	8.6942	2.45
4	0.475	0.0025	12.467	4.51
6	0.638	0.0028	16.732	6.24
8	0.8625	0.0044	22.6378	6.82

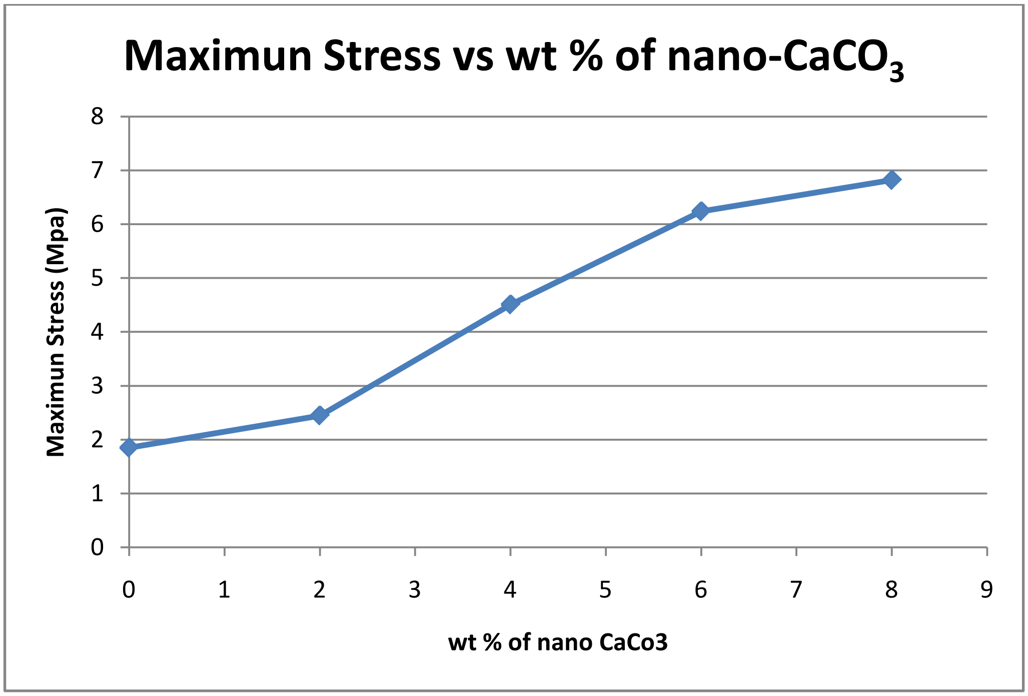


Figure 4.6 : The graph maximum stress vs wt % of nano-CaCO$_3$

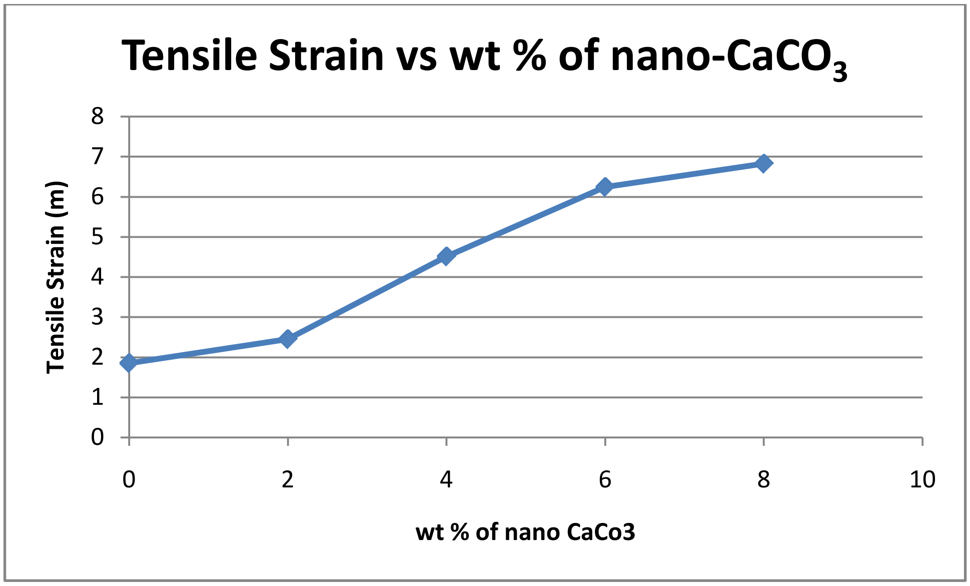


Figure 4.7 : The graph tensile stress vs wt % of nano-$CaCO_3$

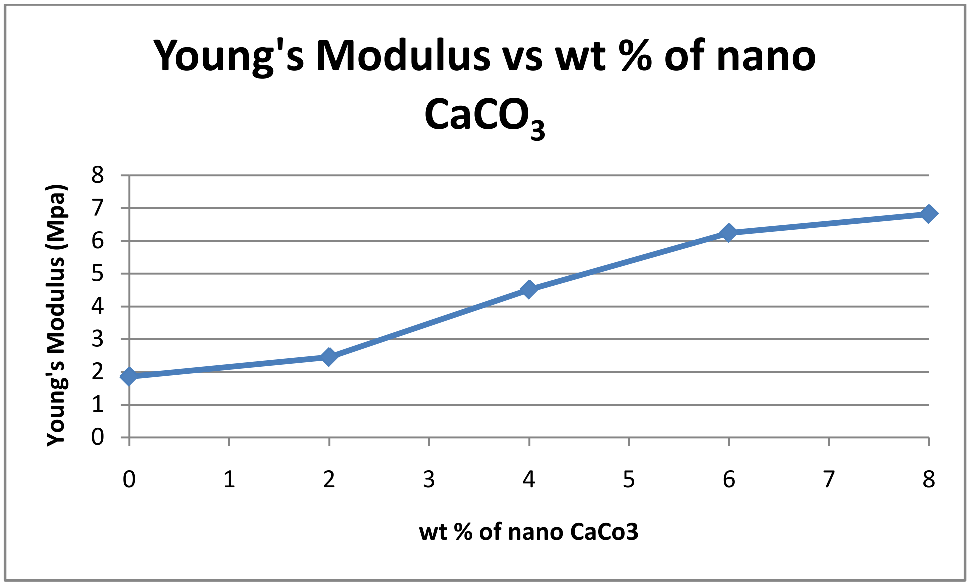


Figure 4.8 : The graph Young’s Modulus vs wt % of nano-$CaCO_3$

The results of the testing are shown by Table 4.1 that is the average value of the tensile tests. There were three samples tested for each type of specimens, which is reinforced to different percentage weight in order to get accurate results. As we can see from Table 4.1, specimen with the highest percentage of nano $CaCo_3$ (8%) has the highest maximum stress (22.64MPa). As the percentage of nano $CaCO_3$ increase, the tensile stress also increases consequently. We can say that the nano $CaCO_3$ is directly proportional to tensile stress. This also can be proven by the trend of the graph plotted as shown in Figure 4.1 where higher the percentage of $CaCO_3$, higher the value of tensile stress. If compare the highest percentage (8%) to the lowest percentage (0%), there is a different about 22.64% which we have proven that nano $CaCO_3$ affect the tensile strength of the material.

For the second graph as shown in Figure 4.7 that been plotted for tensile tests results, the graph tensile strain versus percentage of nano $CaCO_3$ shown trend that almost similar to tensile stress versus wt% of nano $CaCO_3$ has positive slope. As the nano $CaCO_3$ is increase, the tensile strain is increase. Hence, we can say that tensile strain is directly proportional to the nano $CaCO_3$. The highest strain that we got from the tensile is 0.0044 m that from the highest value of wt % nano $CaCO_3$. This is due to highest nano $CaCO_3$ specimen behave more ductile that causing it can elongate longer compare to others specimens. And for the last graph (Figure 4.8) that Young's Modulus versus nano $CaCO_3$, the trend of the graph is similar with the first two that we plotted. It shown that as nano $CaCo_3$ increase, the Young's Modulus will increased. This can be proven by equation of Young's Modulus where the Young's Modulus is inversely proportional to tensile strain.

As a conclusion, as the percentage of nano $CaCO_3$ increase, the tensile stress and tensile strain also increase meanwhile the Young's Modulus will increase. As the strain is decrease, it means that the material become stiffer and brittle.

4.2 Charpy Test Result

Charpy test was conducted to determine the amount energy that has been absorbed by the different percentage of nano Caco$_3$. Table 4.2 shows the result of the energy absorbed with different percentage specimens using pendulum impact test machine. From the Table 4.2, we noticed that the highest percentage weight of nano CaCO$_3$ has the highest energy absorbed that is 0.80J meanwhile the lowest energy absorbed by the specimen is 0.36J that been absorbed by specimen with 0% weight nano CaCO$_3$. The reason why the specimen absorbed more energy due to they have a lots of small spherulites with a lot of grain boundaries. This is because the spherulites do not have the chance to grow due to increasing of nano CaCO$_3$, make their size is small. As the specimens have a lot of small grain boundaries, it hard for the crack to propagate due to grain boundaries impede the crack. Hence, it needed bigger energy to break if compared to the one with lower nano CaCO$_3$. As a conclusion, the amount of energy absorbed by the specimens can be used to measure the fracture toughness of the material and the yield strength.

Table 4.2 : Energy Absorbed vs Nano CaCO$_3$ (%)

Nano CaCO$_3$ (%)	**Energy absorbed (Joule) by pendulum impact test**			
	Sample 1	**Sample 2**	**Sample 3**	**Average**
0	0.4	0.4	0.3	0.36
2	0.5	0.4	0.5	0.47
4	0.5	0.5	0.6	0.53
6	0.7	0.5	0.7	0.63
8	0.8	0.9	0.7	0.80

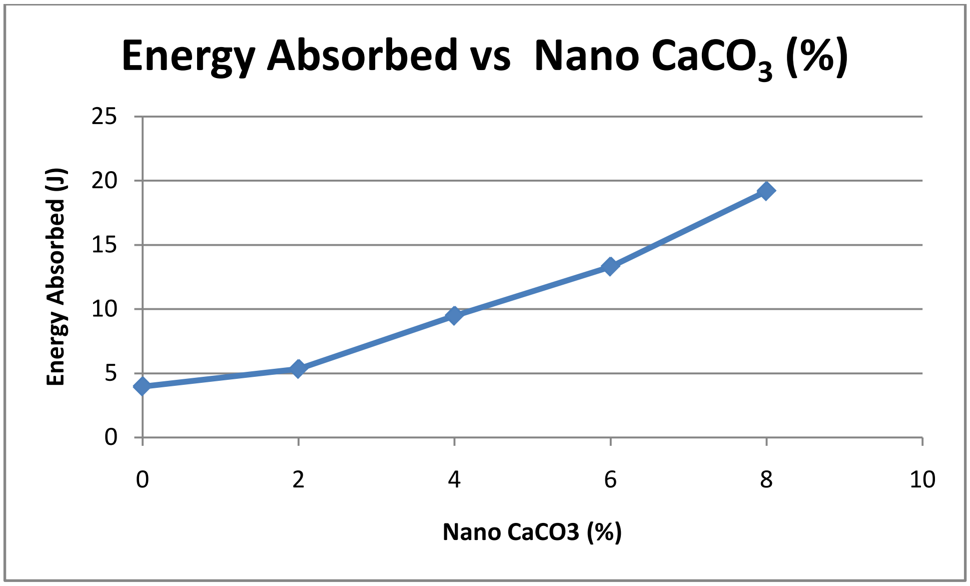


Figure 4.9 : The graph energy absorbed versus nano $CaCO_3$

If a specimen fails with shear lips or jagged edges, it means that the fracture is ductile meanwhile if the specimen fails on flat plat, it means the fracture is brittle. By comparing the shape of failure of the specimen will give us estimation of the failure mode for our Charpy specimens. For Figure 4.9(e), the specimen that have the highest percentage of nano $CaCo_3$, the fracture shows jagged edge and not a straight line. This is because it was not easy to break the specimen, hence it needed more energy to fracture it compare for lower percentage of nano $CaCO_3$ specimens . Hence the specimen behave as ductile. As the percentage of nano $CaCo_3$ increase, the energy needed are higher to break the specimen. As for the specimens with lower percentage of nano $CaCo_3$, from Figure 4.9(a) to Figure 4.9(d), we can see the fracture mode is in straight line or flat plane at failure area. The straight line or flat plane means that the fracture needed a small energy to propagate the crack to fracture and the specimens is weak. For the specimen with lower percentage of nano $CaCO_3$ will behave like this.

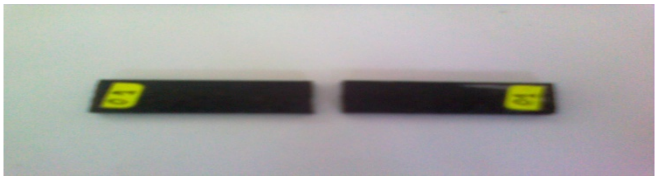


Figure 4.9(a) Charpy failure mode with 0% of nano $CaCO_3$

Figure 4.9(b) Charpy failure mode with 2% of nano $CaCO_3$

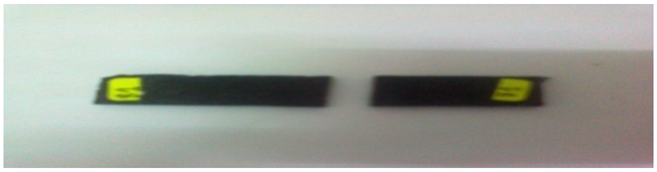


Figure 4.9(c) Charpy failure mode with 4 % of nano $CaCO_3$

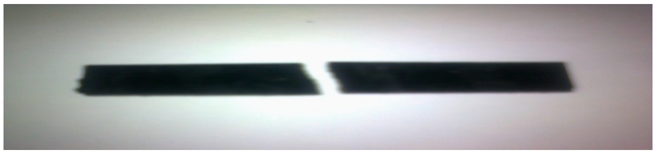


Figure 4.9(d) Charpy failure mode with 6 % of nano $CaCO_3$

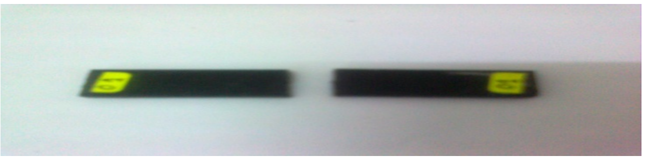


Figure 4.9(e) Charpy failure mode with 8 % of nano $CaCO_3$

4.3 Microhardness Test

Table below shows the microhardness of the samples. Based from figure below, at each specimen, the values of hardness increase when the percentages of nano $CaCO_3$ increase. The highest hardness occurs at 8% of nano $CaCO_3$ and 0% nano $CaCo_3$ have the lowest amount of hardness.

Based from the trend obtained from results, it shows that the increasing of nano $CaCO_3$ gives high value of hardness. The trend is applicable for all sample. For example sample of 8% weight gives the maximum value for each ratio. Hence, it can be conclude that the increased of percentage of Nano $CaCO_3$ will increased the hardness of sample produced.

Table 4.3 : Microhardness vs Nano-$CaCO_3$ (%)

Nano CaCo3 (%)	**Microhardness (Hv)**			
	Sample 1	**Sample 2**	**Sample 3**	**Average**
0	4.3	3.7	3.9	3.97
2	4.2	5.7	6.1	5.33
4	8.3	9.2	10.9	9.47
6	13.3	14.5	12.1	13.3
8	17.8	20.0	19.7	19.17

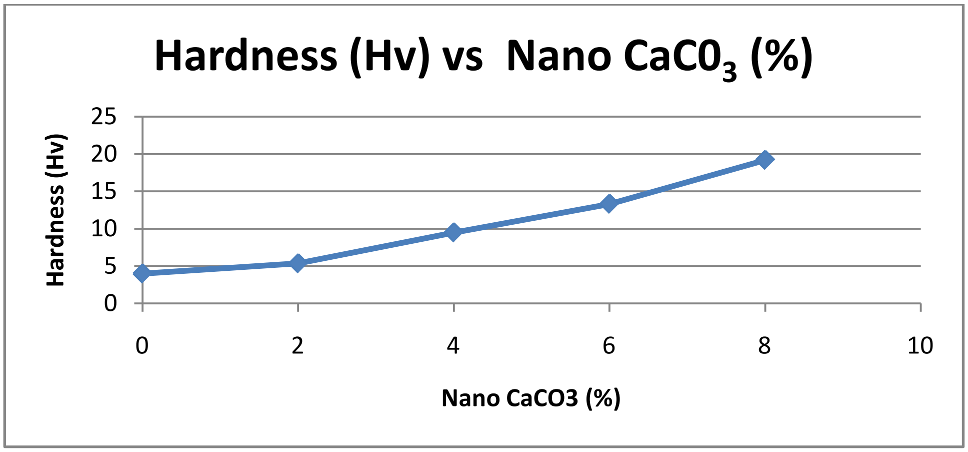


Figure 4.10 The graph value of microhardness versus wt% of nano $CaCO_3$

4.4 Specimen's Microstructure Study

Different percentage of nano $CaCO_3$ have caused variation of degree of crystallinity and appearance samples due to different structural morphology formed. The structural morphology that is changes are the spehrulites which have different sizes due to different percentage of nano $CaCO_3$. This morphology structure change will be discussed in this section and how this change affecting the mechanical properties of the specimens. The spehrulites structure can be assessed by using scanning electron microscope (SEM).

As a result, the specimens that have higher mechanical properties as more energy needed to fail the specimens. We can see from the result of experiments, the specimens with highest weight percentage of nano $CaCO_3$ show greater tensile and flexural stress and also tensile and flexural strains. Due to fine crystals structure and no weak site, more energy needed to fracture the specimens. As for the Young's Modulus of this specimens, it is lower due to the degree of crystallinity is low.

As for the specimens with lower weight percentage of nano $CaCo_3$ condition, the specimens have big spherulites, and there is greater contraction or shrinkage of the crystalline region during and after cooling which leads to the formation of "retraction voids" between the spherulites boundaries at the tetrahedral junction. This is because as the spherulites grow bigger, they will impinge on each other and form the boundary line. These boundaries become the "weak site" and cracks and voids simultaneously forms in the area are due to shrinkage of the bulk polymer. Hence, the existence of retraction voids between the spherulites boundaries partly causing the weakness of this specimen. As a result, the weakness of the structure is causing the tensile stress and strain needed is lower compare to one with high weight percentage of nano $CaCO_3$. As for the Charpy test result, we can see the amount energy absorbed by the specimens are lower compare to the one with increasing nano $CaCO_3$. This is because of the existence of the voids causing lesser energy needed to break the weak sites such micro cracks and voids. For this specimen with lower weight percentage of nano $CaCO_3$ has higher value of Young's Modulus due to have high percentage of degree of crystallinity. As the degree of crystallinity higher, the material will have higher stiffness. Hence, it will have higher Young's Modulus value if compared to highest percentage of nano $CaCO_3$ specimen.

For the lower weight percentage of nano $CaCO_3$, it will have longer crystallization time causing more time for the segregation of "impurities" such as low molecular weight species accumulate between the spherulite boundaries. This high concentration of the impurities likely to accumulate near the retraction voids. The combination of both voids and "impurities" will weaken the structure of the material. The voids are the major reason of inter-spherulatic fracture of large –spherulite specimens. This is because the voids are the first reason cracks been initiated in the structure, and the crack will propagate to where boundary regions are weak. Hence it cause the lower in certain mechanical properties compare to increasing nano $CaCO_3$ specimens.

As we increase the nano $CaCO_3$, we can see improvement in impact strength of the specimens compare the one without calcium carbonate fillers that been conducted by Ling Yih Lii, 2009. By increasing the weight percent of nano $CaCo_3$ to the epoxy composite resulted in a large number of nucleuses and caused large number of spherulites in the limited space. The calcium carbonate nanoparticles are an effective nucleating agent that causes the absence of detectable spherulites. These nanoparticles introduce massive number of stress concentration sites in the matrix and promote cavitation at the particle –matrix boundary when loaded [Chi Ming-Chan, 2002]. The cavities in turn release the plastic constraints and trigger large-scale plastic deformation of the matrix which resulting need higher energy to fracture it compare to epoxy composite without calcium carbonate fillers. As for the tensile stress improvement due to addition of calcium carbonate fillers, there is bonding happens between the epoxy composite. The bond’s strength is good, hence causing higher energy to break the bond. This is resulting higher tensile stress needed.

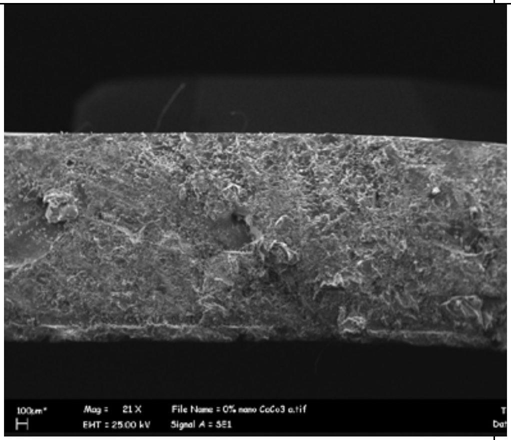
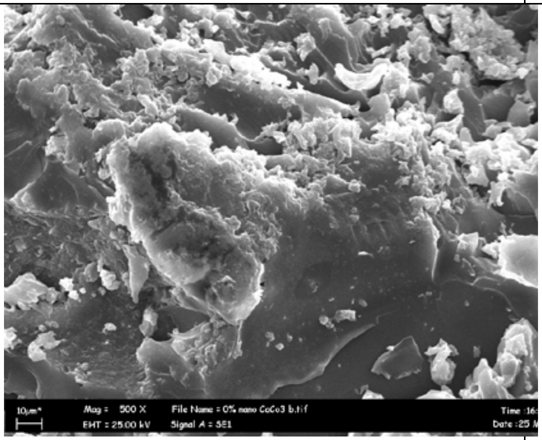
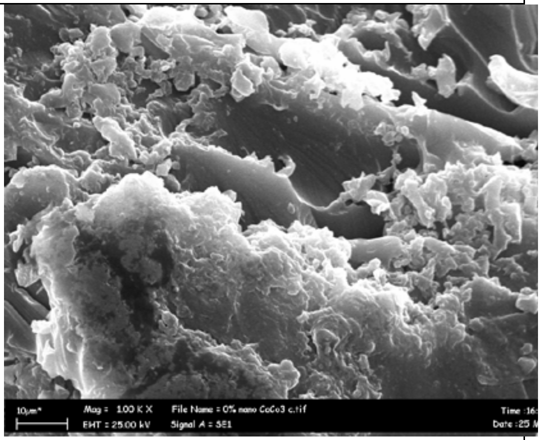
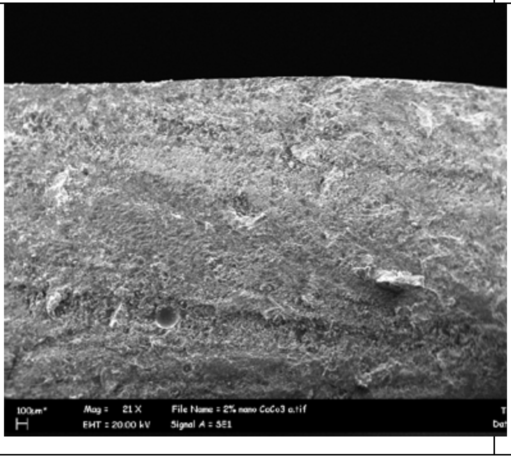
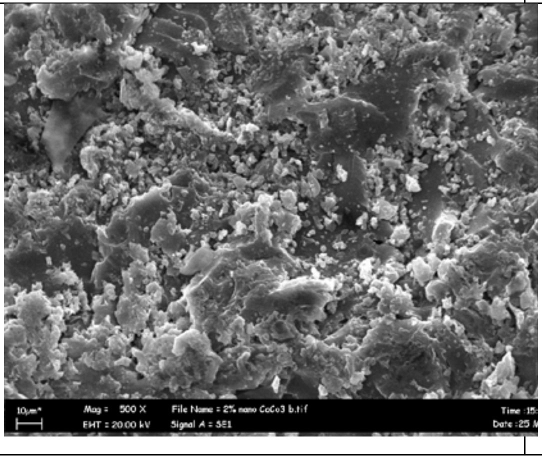
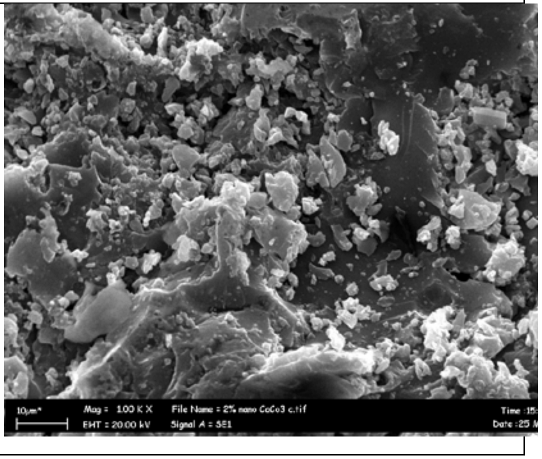
<table>
<tr><th rowspan="2">SAMPLE (wt% of $CaCO_3$)</th><th colspan="3">MAGNIFICATION</th></tr>
<tr><th>21 X</th><th>500 X</th><th>1.00 K X</th></tr>
<tr><td>0%</td><td> </td><td> </td><td> </td></tr>
<tr><td>2%</td><td> </td><td> </td><td> </td></tr>
</table>

Figure 4.11 : Scanning Electron Micrograph of tensile Fractured Surface of 0% and 2 % weight of nano-$CaCO_3$

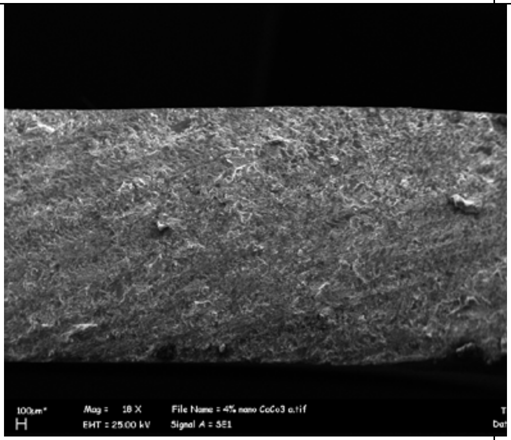
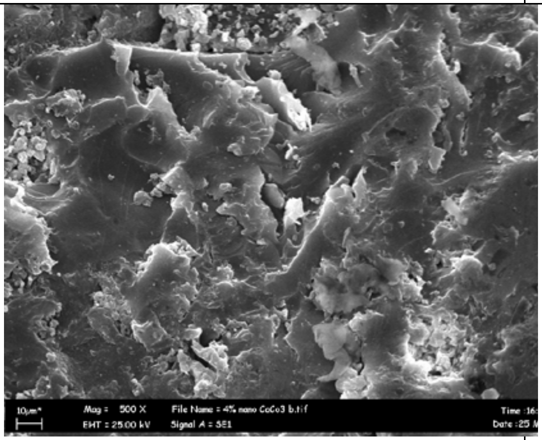
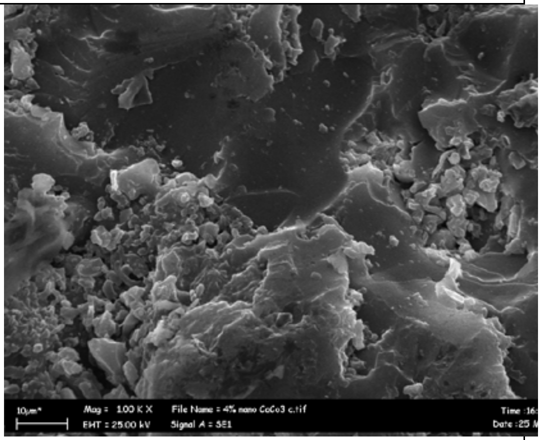
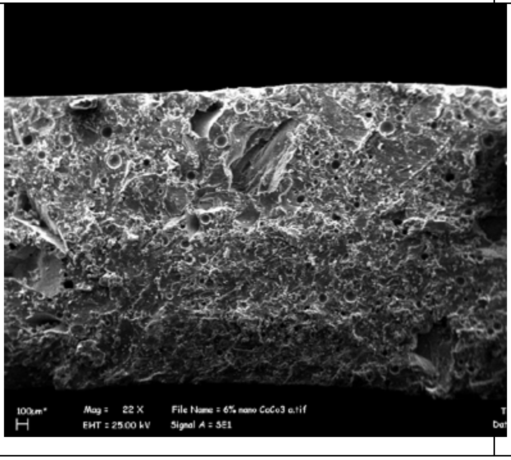
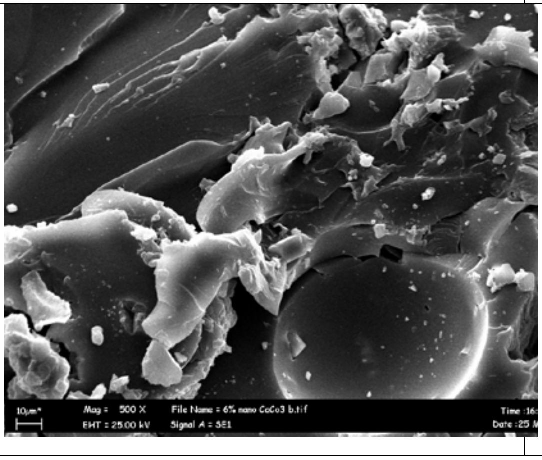
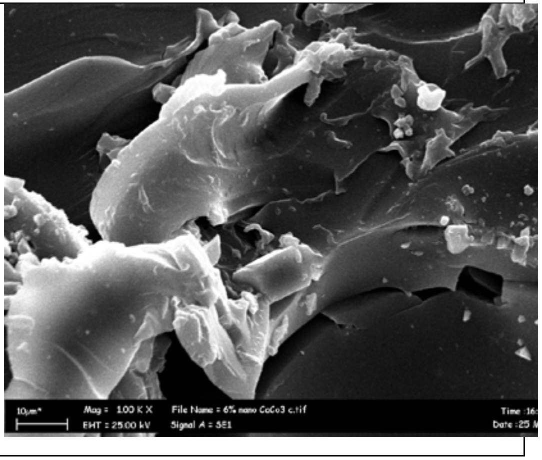
SAMPLE (wt% of CaCO$_3$)	**MAGNIFICATION**		
	21 X	**500 X**	**1.00 K X**
4%			
6%			

Figure 4.12 : Scanning Electron Micrograph of tensile Fractured Surface of 4% and 6 % weight of nano-CaCO$_3$

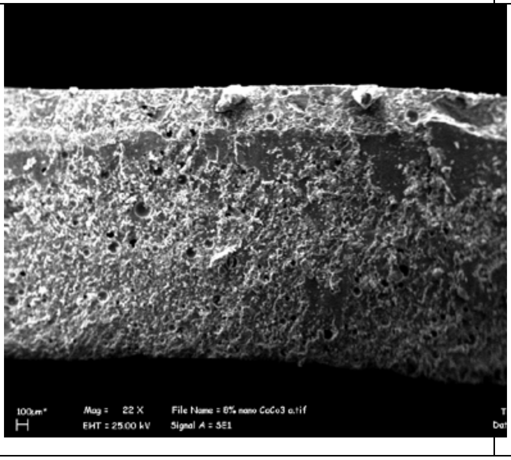
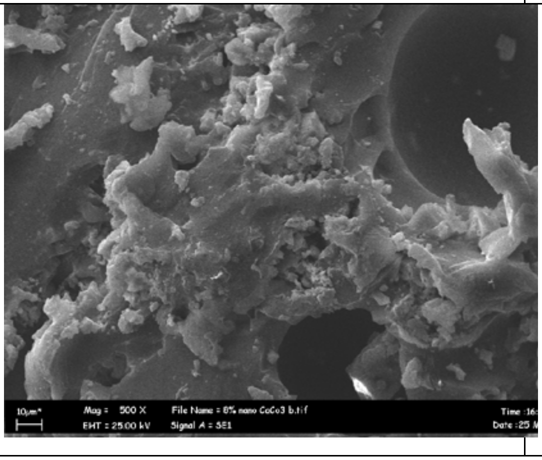
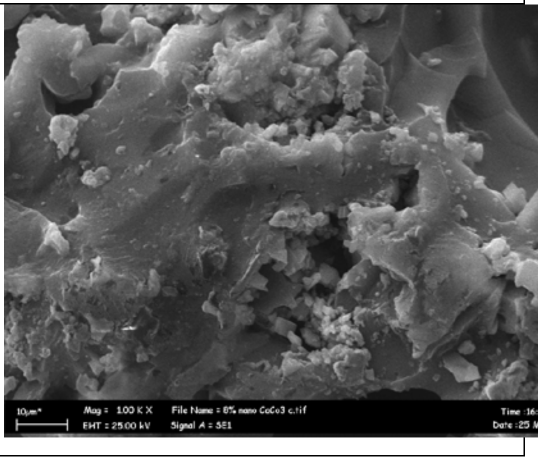
SAMPLE (wt% of CaCO3)	**MAGNIFICATION**		
	21 X	**500 X**	**1.00 K X**
8%			

Figure 4.13 : Scanning Electron Micrograph of tensile Fractured Surface of 8 % weight of nano-$CaCO_3$

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.0 CONCLUSIONS

After all the testing and result analysis, it can be concluded that the objective to determine the effect of nano-$CaCO_3$ to mechanical properties of epoxy composite reinforced recycle rubber has been achieved. All the mechanical properties of these specimens were determine by conducting the tensile test, impact test, Scanning Electron Microscope (SEM) and microhardness test. The effects of nano-$CaCO_3$ towards mechanical properties of epoxy composite can be explained by the analysis of the microstructure.

The whole experiment and the study effect of nano- $CaCO_3$ towards mechanical properties can be summarized as below:

1. The tensile stress of polymer matrix composite are been affected by the nano $CaCo_3$. Higher nano $CaCo_3$ produced higher tensile stress and tensile strain. The maximum stress obtained at higher nano $CaCo_3$ is 22.64 Mpa

2. The tensile strain increase as the nano-$CaCo_3$ is increase.
   Material behaves more ductile as the nano-$CaCo_3$ is increase.

3. The tensile strain is decrease as nano $CaCo_3$ increase. This means the brittle increase as nano $CaCo_3$ is lower.

4. For the Charpy test, more energy been absorbed by specimen with high nano $CaCo_3$. The most energy absorbed is by the highest nano $CaCo_3$ (8%) that is 0.8J. This is due to higher energy needed as the structure has very fine crystal structure with no weak site.

5. As we increased the nano $CaCo_3$ to the epoxy composite, certain mechanical properties such as and impact strength are been improved if compared to pure composites samples.

5.1 RECOMMENDATIONS

To further the investigation of the mechanical properties , some modifications can be suggested to improve the understandings of such material hence lots of study to improve its properties can be done in the future. Thus, some recommendations were suggested for this study as follow:

1. This study only uses 3 mm thickness of specimen to obtain the material’s properties. Hence, for the future study, the specimens can be prepared with different thickness.

2. This study just added 0-8% of nano calcium carbonate fillers to the epoxy composite, for the future study specimens can be prepared with various percentage of fillers and study the effects to the mechanical properties.

3. Insert different types of fillers into the epoxy composite and study the effects on the mechanical properties.

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Gantt Chart of PSM 1

No	Task	Week																
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
1	Selection of PSM title	▨																
		🟥																
2	PSM title selected and filled the form	▨																
		🟥	🟥															
3	Research, study and understand the synopsis of title	▨	▨	▨														
		🟥	🟥	🟥														
4	Find all related informations, journal and references book	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨			
		🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥
5	Discuss the objectives and scopes with supervisor			▨														
				🟥	🟥													
6	Rework and make a correction for objectives and scopes			▨	▨													
				🟥	🟥	🟥												
7	Meet and discuss the progress of PSM with CO supervisor				▨	▨												
					🟥	🟥	🟥											
8	Implement introduction and literature review					▨	▨	▨										
						🟥	🟥	🟥	🟥	🟥								
9	Review the introduction and literature review by supervisor								▨	▨	▨	▨						
									🟥	🟥	🟥	🟥	🟥	🟥				
10	Make a flow chart of method for preparing specimens								▨	▨	▨							
										🟥	🟥	🟥	🟥					
11	Implement the methodology chapter										▨	▨	▨	▨				
												🟥	🟥	🟥				
12	Review methodology by supervisor											▨	▨					
													🟥	🟥				
13	Make a correction for methodology chapter											▨	▨	▨				
													🟥	🟥				
14	Complete the report and submit to supervisor													▨	▨			
															🟥	🟥		
15	Preparation for Oral Presentation (Power Point Slides)															▨	▨	▨
16	Presentation PSM 1																	▨

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▨ Planning

		Gantt Chart of PSM II																				
No	**Task**	**Dec 2010**				**Week - 2011**																
		1	2	3	4	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
1	Seek for raw material	🟨	🟨	🟨	🟨	🟨	🟨															
2	Raw material preparation						🟨	🟨														
3	Mold Preparation							🟨	🟨													
4	Sample Preparation									🟨	🟨	🟨	🟨									
5	Tensile Test													🟨								
6	Tensile Test														🟨							
7	Pendulum Impact Test															🟨						
8	Microhardness Test															🟨	🟨					
9	hardness Test																🟨	🟨				
10	SEM Analysis																	🟨	🟨			
11	Interpret data/ results									🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨			
12	Implement the results and discussion									🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨		
13	Review results and discussion by supervisor															🟨	🟨	🟨	🟨	🟨	🟨	🟨
14	Complete the report and submit to supervisor																			🟨	🟨	🟨
15	Preparation for Oral Presentation (Power Point Slides)																				🟨	🟨
16	Presentation PSM II																					🟨

🟨 Actual

🟥 Planning

Designation: D 638 – 00

An American National Standard

Standard Test Method for Tensile Properties of Plastics[1]

This standard is issued under the fixed designation D 638; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This test method covers the determination of the tensile properties of unreinforced and reinforced plastics in the form of standard dumbbell-shaped test specimens when tested under defined conditions of pretreatment, temperature, humidity, and testing machine speed.

1.2 This test method can be used for testing materials of any thickness up to 14 mm (0.55 in.). However, for testing specimens in the form of thin sheeting, including film less than 1.0 mm (0.04 in.) in thickness, Test Methods D 882 is the preferred test method. Materials with a thickness greater than 14 mm (0.55 in.) must be reduced by machining.

1.3 This test method includes the option of determining Poisson's ratio at room temperature.

NOTE 1—This test method and ISO 527-1 are technically equivalent.

NOTE 2—This test method is not intended to cover precise physical procedures. It is recognized that the constant rate of crosshead movement type of test leaves much to be desired from a theoretical standpoint, that wide differences may exist between rate of crosshead movement and rate of strain between gage marks on the specimen, and that the testing speeds specified disguise important effects characteristic of materials in the plastic state. Further, it is realized that variations in the thicknesses of test specimens, which are permitted by these procedures, produce variations in the surface-volume ratios of such specimens, and that these variations may influence the test results. Hence, where directly comparable results are desired, all samples should be of equal thickness. Special additional tests should be used where more precise physical data are needed.

NOTE 3—This test method may be used for testing phenolic molded resin or laminated materials. However, where these materials are used as electrical insulation, such materials should be tested in accordance with Test Methods D 229 and Test Method D 651.

NOTE 4—For tensile properties of resin-matrix composites reinforced with oriented continuous or discontinuous high modulus >20-GPa ($>3.0 \times 10^6$-psi) fibers, tests shall be made in accordance with Test Method D 3039/D 3039M.

1.4 Test data obtained by this test method are relevant and appropriate for use in engineering design.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 229 Test Methods for Rigid Sheet and Plate Materials Used for Electrical Insulation[2]
D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension[3]
D 618 Practice for Conditioning Plastics for Testing[4]
D 651 Test Method for Tensile Strength of Molded Electrical Insulating Materials[5]
D 882 Test Methods for Tensile Properties of Thin Plastic Sheeting[4]
D 883 Terminology Relating to Plastics[4]
D 1822 Test Method for Tensile-Impact Energy to Break Plastics and Electrical Insulating Materials[4]
D 3039/D 3039M Test Method for Tensile Properties of Polymer Matrix Composite Materials[6]
D 4000 Classification System for Specifying Plastic Materials[7]
D 4066 Specification for Nylon Injection and Extrusion Materials[7]
D 5947 Test Methods for Physical Dimensions of Solid Plastic Specimens[8]
E 4 Practices for Force Verification of Testing Machines[9]
E 83 Practice for Verification and Classification of Extensometer Systems[9]
E 132 Test Method for Poisson's Ratio at Room Temperature[9]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[10]

2.2 *ISO Standard:*

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[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D 20.10 on Mechanical Properties.

Current edition approved Dec. 10, 2000. Published February 2001. Originally published as D 638 – 41 T. Last previous edition D 638 – 99.

[2] *Annual Book of ASTM Standards*, Vol 10.01.
[3] *Annual Book of ASTM Standards*, Vol 09.01.
[4] *Annual Book of ASTM Standards*, Vol 08.01.
[5] Discontinued; see *1994 Annual Book of ASTM Standards*, Vol 10.01.
[6] *Annual Book of ASTM Standards*, Vol 15.03.
[7] *Annual Book of ASTM Standards*, Vol 08.02.
[8] *Annual Book of ASTM Standards*, Vol 08.03.
[9] *Annual Book of ASTM Standards*, Vol 03.01.
[10] *Annual Book of ASTM Standards*, Vol 14.02.

***A Summary of Changes section appears at the end of this standard.**

ISO 527-1 Determination of Tensile Properties[11]

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in Terminology D 883 and Annex A2.

4. Significance and Use

4.1 This test method is designed to produce tensile property data for the control and specification of plastic materials. These data are also useful for qualitative characterization and for research and development. For many materials, there may be a specification that requires the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D 4000 lists the ASTM materials standards that currently exist.

4.2 Tensile properties may vary with specimen preparation and with speed and environment of testing. Consequently, where precise comparative results are desired, these factors must be carefully controlled.

4.2.1 It is realized that a material cannot be tested without also testing the method of preparation of that material. Hence, when comparative tests of materials per se are desired, the greatest care must be exercised to ensure that all samples are prepared in exactly the same way, unless the test is to include the effects of sample preparation. Similarly, for referee purposes or comparisons within any given series of specimens, care must be taken to secure the maximum degree of uniformity in details of preparation, treatment, and handling.

4.3 Tensile properties may provide useful data for plastics engineering design purposes. However, because of the high degree of sensitivity exhibited by many plastics to rate of straining and environmental conditions, data obtained by this test method cannot be considered valid for applications involving load-time scales or environments widely different from those of this test method. In cases of such dissimilarity, no reliable estimation of the limit of usefulness can be made for most plastics. This sensitivity to rate of straining and environment necessitates testing over a broad load-time scale (including impact and creep) and range of environmental conditions if tensile properties are to suffice for engineering design purposes.

NOTE 5—Since the existence of a true elastic limit in plastics (as in many other organic materials and in many metals) is debatable, the propriety of applying the term “elastic modulus” in its quoted, generally accepted definition to describe the “stiffness” or “rigidity” of a plastic has been seriously questioned. The exact stress-strain characteristics of plastic materials are highly dependent on such factors as rate of application of stress, temperature, previous history of specimen, etc. However, stress-strain curves for plastics, determined as described in this test method, almost always show a linear region at low stresses, and a straight line drawn tangent to this portion of the curve permits calculation of an elastic modulus of the usually defined type. Such a constant is useful if its arbitrary nature and dependence on time, temperature, and similar factors are realized.

4.4 *Poisson’s Ratio*—When uniaxial tensile force is applied to a solid, the solid stretches in the direction of the applied force (axially), but it also contracts in both dimensions lateral to the applied force. If the solid is homogeneous and isotropic, and the material remains elastic under the action of the applied force, the lateral strain bears a constant relationship to the axial strain. This constant, called Poisson’s ratio, is defined as the negative ratio of the transverse (negative) to axial strain under uniaxial stress.

4.4.1 Poisson’s ratio is used for the design of structures in which all dimensional changes resulting from the application of force need to be taken into account and in the application of the generalized theory of elasticity to structural analysis.

NOTE 6—The accuracy of the determination of Poisson’s ratio is usually limited by the accuracy of the transverse strain measurements because the percentage errors in these measurements are usually greater than in the axial strain measurements. Since a ratio rather than an absolute quantity is measured, it is only necessary to know accurately the relative value of the calibration factors of the extensometers. Also, in general, the value of the applied loads need not be known accurately.

5. Apparatus

5.1 *Testing Machine*—A testing machine of the constant-rate-of-crosshead-movement type and comprising essentially the following:

5.1.1 *Fixed Member*—A fixed or essentially stationary member carrying one grip.

5.1.2 *Movable Member*—A movable member carrying a second grip.

5.1.3 *Grips*—Grips for holding the test specimen between the fixed member and the movable member of the testing machine can be either the fixed or self-aligning type.

5.1.3.1 Fixed grips are rigidly attached to the fixed and movable members of the testing machine. When this type of grip is used extreme care should be taken to ensure that the test specimen is inserted and clamped so that the long axis of the test specimen coincides with the direction of pull through the center line of the grip assembly.

5.1.3.2 Self-aligning grips are attached to the fixed and movable members of the testing machine in such a manner that they will move freely into alignment as soon as any load is applied so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. The specimens should be aligned as perfectly as possible with the direction of pull so that no rotary motion that may induce slippage will occur in the grips; there is a limit to the amount of misalignment self-aligning grips will accommodate.

5.1.3.3 The test specimen shall be held in such a way that slippage relative to the grips is prevented insofar as possible. Grip surfaces that are deeply scored or serrated with a pattern similar to those of a coarse single-cut file, serrations about 2.4 mm (0.09 in.) apart and about 1.6 mm (0.06 in.) deep, have been found satisfactory for most thermoplastics. Finer serrations have been found to be more satisfactory for harder plastics, such as the thermosetting materials. The serrations should be kept clean and sharp. Breaking in the grips may occur at times, even when deep serrations or abraded specimen surfaces are used; other techniques must be used in these cases.

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[11] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

Other techniques that have been found useful, particularly with smooth-faced grips, are abrading that portion of the surface of the specimen that will be in the grips, and interposing thin pieces of abrasive cloth, abrasive paper, or plastic, or rubber-coated fabric, commonly called hospital sheeting, between the specimen and the grip surface. No. 80 double-sided abrasive paper has been found effective in many cases. An open-mesh fabric, in which the threads are coated with abrasive, has also been effective. Reducing the cross-sectional area of the specimen may also be effective. The use of special types of grips is sometimes necessary to eliminate slippage and breakage in the grips.

5.1.4 *Drive Mechanism*—A drive mechanism for imparting to the movable member a uniform, controlled velocity with respect to the stationary member, with this velocity to be regulated as specified in Section 8.

5.1.5 *Load Indicator*—A suitable load-indicating mechanism capable of showing the total tensile load carried by the test specimen when held by the grips. This mechanism shall be essentially free of inertia lag at the specified rate of testing and shall indicate the load with an accuracy of ±1 % of the indicated value, or better. The accuracy of the testing machine shall be verified in accordance with Practices E 4.

NOTE 7—Experience has shown that many testing machines now in use are incapable of maintaining accuracy for as long as the periods between inspection recommended in Practices E 4. Hence, it is recommended that each machine be studied individually and verified as often as may be found necessary. It frequently will be necessary to perform this function daily.

5.1.6 The fixed member, movable member, drive mechanism, and grips shall be constructed of such materials and in such proportions that the total elastic longitudinal strain of the system constituted by these parts does not exceed 1 % of the total longitudinal strain between the two gage marks on the test specimen at any time during the test and at any load up to the rated capacity of the machine.

5.2 *Extension Indicator* (extensometer)—A suitable instrument shall be used for determining the distance between two designated points within the gage length of the test specimen as the specimen is stretched. For referee purposes, the extensometer must be set at the full gage length of the specimen, as shown in Fig. 1. It is desirable, but not essential, that this instrument automatically record this distance, or any change in it, as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. If only the latter is obtained, load-time data must also be taken. This instrument shall be essentially free of inertia at the specified speed of testing. Extensometers shall be classified and their calibration periodically verified in accordance with Practice E 83.

5.2.1 *Modulus-of-Elasticity Measurements*—For modulus-of-elasticity measurements, an extensometer with a maximum strain error of 0.0002 mm/mm (in./in.) that automatically and continuously records shall be used. An extensometer classified by Practice E 83 as fulfilling the requirements of a B-2 classification within the range of use for modulus measurements meets this requirement.

5.2.2 *Low-Extension Measurements*—For elongation-at-yield and low-extension measurements (nominally 20 % or less), the same above extensometer, attenuated to 20 % extension, may be used. In any case, the extensometer system must meet at least Class C (Practice E 83) requirements, which include a fixed strain error of 0.001 strain or ±1.0 % of the indicated strain, whichever is greater.

5.2.3 *High-Extension Measurements*—For making measurements at elongations greater than 20 %, measuring techniques with error no greater than ±10 % of the measured value are acceptable.

5.2.4 *Poisson's Ratio*—Bi-axial extensometer or axial and transverse extensometers capable of recording axial strain and transverse strain simultaneously. The extensometers shall be capable of measuring the change in strains with an accuracy of 1 % of the relevant value or better.

NOTE 8—Strain gages can be used as an alternative method to measure axial and transverse strain; however, proper techniques for mounting strain gages are crucial to obtaining accurate data. Consult strain gage suppliers for instruction and training in these special techniques.

5.3 *Micrometers*—Suitable micrometers for measuring the width and thickness of the test specimen to an incremental discrimination of at least 0.025 mm (0.001 in.) should be used. All width and thickness measurements of rigid and semirigid plastics may be measured with a hand micrometer with ratchet. A suitable instrument for measuring the thickness of nonrigid test specimens shall have: *(1)* a contact measuring pressure of 25 ± 2.5 kPa (3.6 ± 0.36 psi), *(2)* a movable circular contact foot 6.35 ± 0.025 mm (0.250 ± 0.001 in.) in diameter, and *(3)* a lower fixed anvil large enough to extend beyond the contact foot in all directions and being parallel to the contact foot within 0.005 mm (0.0002 in.) over the entire foot area. Flatness of the foot and anvil shall conform to Test Method D 5947.

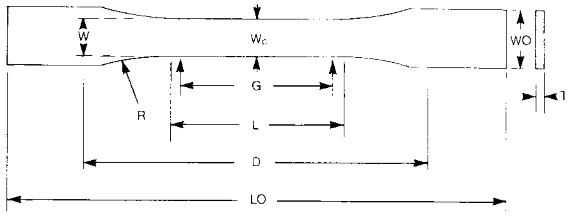
5.3.1 An optional instrument equipped with a circular contact foot 15.88 ± 0.08 mm (0.625 ± 0.003 in.) in diameter is recommended for thickness measuring of process samples or larger specimens at least 15.88 mm in minimum width.

6. Test Specimens

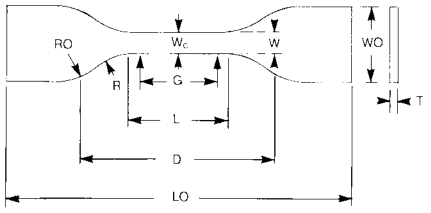
6.1 *Sheet, Plate, and Molded Plastics*:

6.1.1 *Rigid and Semirigid Plastics*—The test specimen shall conform to the dimensions shown in Fig. 1. The Type I specimen is the preferred specimen and shall be used where sufficient material having a thickness of 7 mm (0.28 in.) or less is available. The Type II specimen may be used when a material does not break in the narrow section with the preferred Type I specimen. The Type V specimen shall be used where only limited material having a thickness of 4 mm (0.16 in.) or less is available for evaluation, or where a large number of specimens are to be exposed in a limited space (thermal and environmental stability tests, etc.). The Type IV specimen should be used when direct comparisons are required between materials in different rigidity cases (that is, nonrigid and semirigid). The Type III specimen must be used for all materials with a thickness of greater than 7 mm (0.28 in.) but not more than 14 mm (0.55 in.).

6.1.2 *Nonrigid Plastics*—The test specimen shall conform to the dimensions shown in Fig. 1. The Type IV specimen shall be used for testing nonrigid plastics with a thickness of 4 mm (0.16 in.) or less. The Type III specimen must be used for all materials with a thickness greater than 7 mm (0.28 in.) but not



TYPES I, II, III & IV



TYPE IV

Specimen Dimensions for Thickness, T, mm (in.)[A]

Dimensions (see drawings)	7 (0.28) or under		Over 7 to 14 (0.28 to 0.55), incl	4 (0.16) or under		Tolerances
	Type I	Type II	Type III	Type IV[B]	Type V[C,D]	
W—Width of narrow section[E,F]	13 (0.50)	6 (0.25)	19 (0.75)	6 (0.25)	3.18 (0.125)	±0.5 (±0.02)[B,C]
L—Length of narrow section	57 (2.25)	57 (2.25)	57 (2.25)	33 (1.30)	9.53 (0.375)	±0.5 (±0.02)[C]
WO—Width overall, min[G]	19 (0.75)	19 (0.75)	29 (1.13)	19 (0.75)	...	+ 6.4 (+ 0.25)
WO—Width overall, min[G]	...	...	...	...	9.53 (0.375)	+ 3.18 (+ 0.125)
LO—Length overall, min[H]	165 (6.5)	183 (7.2)	246 (9.7)	115 (4.5)	63.5 (2.5)	no max (no max)
G—Gage length[I]	50 (2.00)	50 (2.00)	50 (2.00)	...	7.62 (0.300)	±0.25 (±0.010)[C]
G—Gage length[I]	...	...	...	25 (1.00)	...	±0.13 (±0.005)
D—Distance between grips	115 (4.5)	135 (5.3)	115 (4.5)	65 (2.5)[J]	25.4 (1.0)	±5 (±0.2)
R—Radius of fillet	76 (3.00)	76 (3.00)	76 (3.00)	14 (0.56)	12.7 (0.5)	±1 (±0.04)[C]
RO—Outer radius (Type IV)	...	...	...	25 (1.00)	...	±1 (±0.04)

[A] Thickness, T, shall be 3.2± 0.4 mm (0.13 ± 0.02 in.) for all types of molded specimens, and for other Types I and II specimens where possible. If specimens are machined from sheets or plates, thickness, T, may be the thickness of the sheet or plate provided this does not exceed the range stated for the intended specimen type. For sheets of nominal thickness greater than 14 mm (0.55 in.) the specimens shall be machined to 14 ± 0.4 mm (0.55 ± 0.02 in.) in thickness, for use with the Type III specimen. For sheets of nominal thickness between 14 and 51 mm (0.55 and 2 in.) approximately equal amounts shall be machined from each surface. For thicker sheets both surfaces of the specimen shall be machined, and the location of the specimen with reference to the original thickness of the sheet shall be noted. Tolerances on thickness less than 14 mm (0.55 in.) shall be those standard for the grade of material tested.

[B] For the Type IV specimen, the internal width of the narrow section of the die shall be 6.00 ± 0.05 mm (0.250± 0.002 in.). The dimensions are essentially those of Die C in Test Methods D 412.

[C] The Type V specimen shall be machined or die cut to the dimensions shown, or molded in a mold whose cavity has these dimensions. The dimensions shall be:
W = 3.18 ± 0.03 mm (0.125 ± 0.001 in.),
L = 9.53 ± 0.08 mm (0.375 ± 0.003 in.),
G = 7.62 ± 0.02 mm (0.300 ± 0.001 in.), and
R = 12.7 ± 0.08 mm (0.500 ± 0.003 in.).
The other tolerances are those in the table.

[D] Supporting data on the introduction of the L specimen of Test Method D 1822 as the Type V specimen are available from ASTM Headquarters. Request RR:D20-1038.

[E] The width at the center W_c shall be +0.00 mm, −0.10 mm (+0.000 in., −0.004 in.) compared with width W at other parts of the reduced section. Any reduction in W at the center shall be gradual, equally on each side so that no abrupt changes in dimension result.

[F] For molded specimens, a draft of not over 0.13 mm (0.005 in.) may be allowed for either Type I or II specimens 3.2 mm (0.13 in.) in thickness, and this should be taken into account when calculating width of the specimen. Thus a typical section of a molded Type I specimen, having the maximum allowable draft, could be as follows:

[G] Overall widths greater than the minimum indicated may be desirable for some materials in order to avoid breaking in the grips.

[H] Overall lengths greater than the minimum indicated may be desirable either to avoid breaking in the grips or to satisfy special test requirements.

[I] Test marks or initial extensometer span.

[J] When self-tightening grips are used, for highly extensible polymers, the distance between grips will depend upon the types of grips used and may not be critical if maintained uniform once chosen.

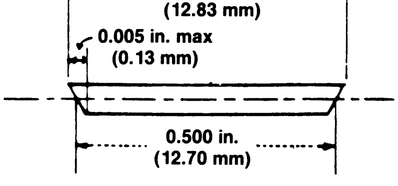
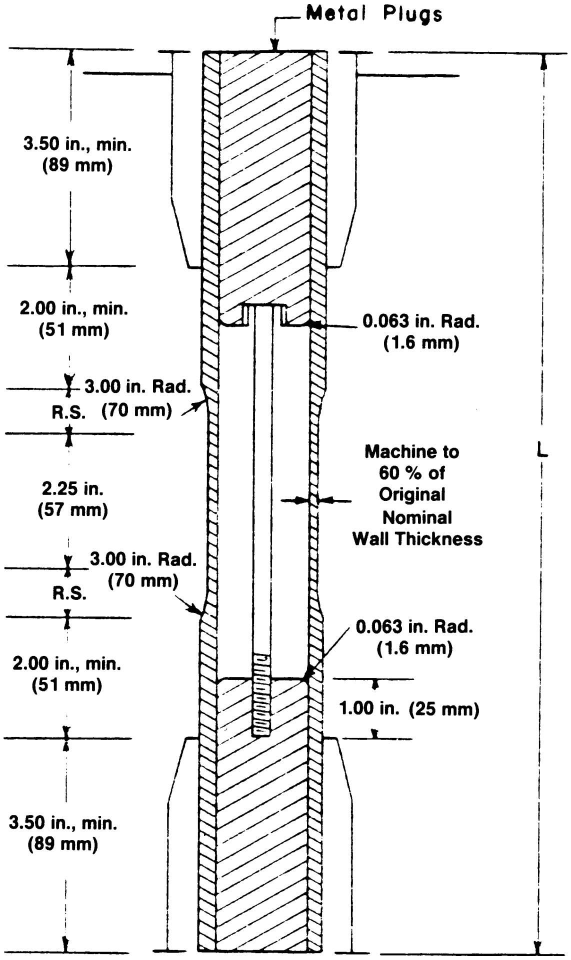


FIG. 1 Tension Test Specimens for Sheet, Plate, and Molded Plastics

more than 14 mm (0.55 in.).

6.1.3 *Reinforced Composites*—The test specimen for reinforced composites, including highly orthotropic laminates, shall conform to the dimensions of the Type I specimen shown in Fig. 1.

6.1.4 *Preparation*—Test specimens shall be prepared by machining operations, or die cutting, from materials in sheet, plate, slab, or similar form. Materials thicker than 14 mm (0.55 in.) must be machined to 14 mm (0.55 in.) for use as Type III specimens. Specimens can also be prepared by molding the material to be tested.



DIMENSIONS OF TUBE SPECIMENS

Nominal Wall Thickness	Length of Radial Sections, 2R.S.	Total Calculated Minimum Length of Specimen	Standard Length, *L*, of Specimen to Be Used for 89-mm (3.5-in.) Jaws[A]
		mm (in.)	
0.79 (1⁄32)	13.9 (0.547)	350 (13.80)	381 (15)
1.2 (3⁄64)	17.0 (0.670)	354 (13.92)	381 (15)
1.6 (1⁄16)	19.6 (0.773)	356 (14.02)	381 (15)
2.4 (3⁄32)	24.0 (0.946)	361 (14.20)	381 (15)
3.2 (1⁄8)	27.7 (1.091)	364 (14.34)	381 (15)
4.8 (3⁄16)	33.9 (1.333)	370 (14.58)	381 (15)
6.4 (1⁄4)	39.0 (1.536)	376 (14.79)	400 (15.75)
7.9 (5⁄16)	43.5 (1.714)	380 (14.96)	400 (15.75)
9.5 (3⁄8)	47.6 (1.873)	384 (15.12)	400 (15.75)
11.1 (7⁄16)	51.3 (2.019)	388 (15.27)	400 (15.75)
12.7 (1⁄2)	54.7 (2.154)	391 (15.40)	419 (16.5)

[A] For other jaws greater than 89 mm (3.5 in.), the standard length shall be increased by twice the length of the jaws minus 178 mm (7 in.). The standard length permits a slippage of approximately 6.4 to 12.7 mm (0.25 to 0.50 in.) in each jaw while maintaining the maximum length of the jaw grip.

FIG. 2 Diagram Showing Location of Tube Tension Test Specimens in Testing Machine

NOTE 9—Test results have shown that for some materials such as glass cloth, SMC, and BMC laminates, other specimen types should be considered to ensure breakage within the gage length of the specimen, as mandated by 7.3.

NOTE 10—When preparing specimens from certain composite laminates such as woven roving, or glass cloth, care must be exercised in cutting the specimens parallel to the reinforcement. The reinforcement will be significantly weakened by cutting on a bias, resulting in lower laminate properties, unless testing of specimens in a direction other than parallel with the reinforcement constitutes a variable being studied.

NOTE 11—Specimens prepared by injection molding may have different tensile properties than specimens prepared by machining or die-cutting because of the orientation induced. This effect may be more pronounced in specimens with narrow sections.

6.2 *Rigid Tubes*—The test specimen for rigid tubes shall be as shown in Fig. 2. The length, *L*, shall be as shown in the table in Fig. 2. A groove shall be machined around the outside of the specimen at the center of its length so that the wall section after machining shall be 60 % of the original nominal wall thickness. This groove shall consist of a straight section 57.2 mm (2.25 in.) in length with a radius of 76 mm (3 in.) at each end joining it to the outside diameter. Steel or brass plugs having diameters such that they will fit snugly inside the tube and having a length equal to the full jaw length plus 25 mm (1 in.) shall be placed in the ends of the specimens to prevent crushing. They can be located conveniently in the tube by separating and supporting them on a threaded metal rod. Details of plugs and test assembly are shown in Fig. 2.

6.3 *Rigid Rods*—The test specimen for rigid rods shall be as shown in Fig. 3. The length, *L*, shall be as shown in the table in Fig. 3. A groove shall be machined around the specimen at the center of its length so that the diameter of the machined portion shall be 60 % of the original nominal diameter. This groove shall consist of a straight section 57.2 mm (2.25 in.) in length with a radius of 76 mm (3 in.) at each end joining it to the outside diameter.

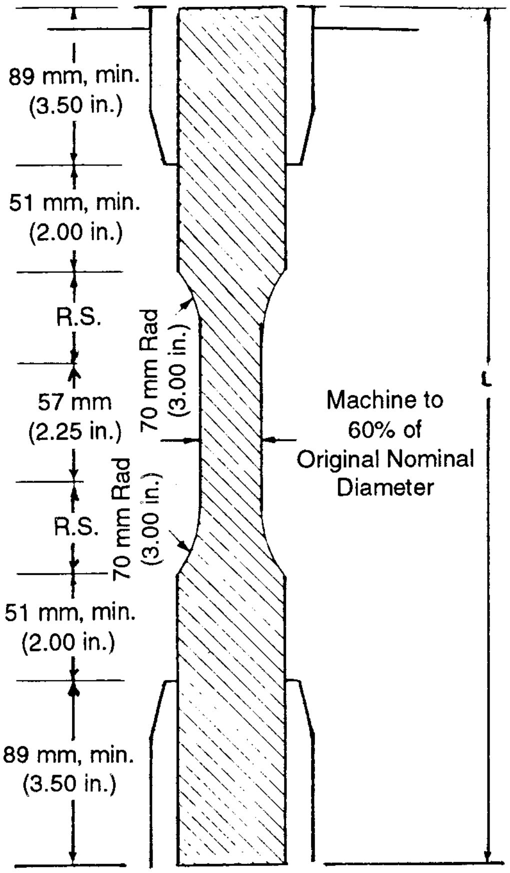
6.4 All surfaces of the specimen shall be free of visible flaws, scratches, or imperfections. Marks left by coarse machining operations shall be carefully removed with a fine file or abrasive, and the filed surfaces shall then be smoothed with abrasive paper (No. 00 or finer). The finishing sanding strokes shall be made in a direction parallel to the long axis of the test specimen. All flash shall be removed from a molded specimen, taking great care not to disturb the molded surfaces. In machining a specimen, undercuts that would exceed the dimensional tolerances shown in Fig. 1 shall be scrupulously avoided. Care shall also be taken to avoid other common machining errors.

6.5 If it is necessary to place gage marks on the specimen, this shall be done with a wax crayon or India ink that will not affect the material being tested. Gage marks shall not be scratched, punched, or impressed on the specimen.

6.6 When testing materials that are suspected of anisotropy, duplicate sets of test specimens shall be prepared, having their long axes respectively parallel with, and normal to, the suspected direction of anisotropy.

7. Number of Test Specimens

7.1 Test at least five specimens for each sample in the case of isotropic materials.



DIMENSIONS OF ROD SPECIMENS

Nominal Diameter	Length of Radial Sections, 2R.S.	Total Calculated Minimum Length of Specimen	Standard Length, *L*, of Specimen to Be Used for 89-mm (3½-in.) Jaws^A
	mm (in.)		
3.2 (1⁄8)	19.6 (0.773)	356 (14.02)	381 (15)
4.7 (3⁄16)	24.0 (0.946)	361 (14.20)	381 (15)
6.4 (1⁄4)	27.7 (1.091)	364 (14.34)	381 (15)
9.5 (3⁄8)	33.9 (1.333)	370 (14.58)	381 (15)
12.7 (1⁄2)	39.0 (1.536)	376 (14.79)	400 (15.75)
15.9 (5⁄8)	43.5 (1.714)	380 (14.96)	400 (15.75)
19.0 (3⁄4)	47.6 (1.873)	384 (15.12)	400 (15.75)
22.2 (7⁄8)	51.5 (2.019)	388 (15.27)	400 (15.75)
25.4 (1)	54.7 (2.154)	391 (15.40)	419 (16.5)
31.8 (1¼)	60.9 (2.398)	398 (15.65)	419 (16.5)
38.1 (1½)	66.4 (2.615)	403 (15.87)	419 (16.5)
42.5 (1¾)	71.4 (2.812)	408 (16.06)	419 (16.5)
50.8 (2)	76.0 (2.993)	412 (16.24)	432 (17)

[A] For other jaws greater than 89 mm (3.5 in.), the standard length shall be increased by twice the length of the jaws minus 178 mm (7 in.). The standard length permits a slippage of approximately 6.4 to 12.7 mm (0.25 to 0.50 in.) in each jaw while maintaining the maximum length of the jaw grip.

FIG. 3 Diagram Showing Location of Rod Tension Test Specimen in Testing Machine

7.2 Test ten specimens, five normal to, and five parallel with, the principle axis of anisotropy, for each sample in the case of anisotropic materials.

7.3 Discard specimens that break at some obvious fortuitous flaw, or that do not break between the predetermined gage marks, and make retests, unless such flaws constitute a variable to be studied.

NOTE 12—Before testing, all transparent specimens should be inspected in a polariscope. Those which show atypical or concentrated strain patterns should be rejected, unless the effects of these residual strains constitute a variable to be studied.

8. Speed of Testing

8.1 Speed of testing shall be the relative rate of motion of the grips or test fixtures during the test. The rate of motion of the driven grip or fixture when the testing machine is running idle may be used, if it can be shown that the resulting speed of testing is within the limits of variation allowed.

8.2 Choose the speed of testing from Table 1. Determine this chosen speed of testing by the specification for the material being tested, or by agreement between those concerned. When the speed is not specified, use the lowest speed shown in Table 1 for the specimen geometry being used, which gives rupture within ½ to 5-min testing time.

8.3 Modulus determinations may be made at the speed selected for the other tensile properties when the recorder response and resolution are adequate.

8.4 Poisson's ratio determinations shall be made at the same speed selected for modulus determinations.

9. Conditioning

9.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be± 1°C (1.8°F) and ±2 % relative humidity.

9.1.1 Note that for some hygroscopic materials, such as nylons, the material specifications (for example, Specification D 4066) call for testing "dry as-molded specimens." Such requirements take precedence over the above routine preconditioning to 50 % relative humidity and require sealing the specimens in water vapor-impermeable containers as soon as molded and not removing them until ready for testing.

TABLE 1 Designations for Speed of Testing^A

Classification^B	Specimen Type	Speed of Testing, mm/min (in./min)	Nominal Strain^C Rate at Start of Test, mm/mm· min (in./in.·min)
Rigid and Semirigid	I, II, III rods and tubes	5 (0.2) ± 25 %	0.1
		50 (2) ± 10 %	1
		500 (20) ± 10 %	10
	IV	5 (0.2) ± 25 %	0.15
		50 (2) ± 10 %	1.5
		500 (20) ± 10 %	15
	V	1 (0.05) ± 25 %	0.1
		10 (0.5) ± 25 %	1
		100 (5)± 25 %	10
Nonrigid	III	50 (2) ± 10 %	1
		500 (20) ± 10 %	10
	IV	50 (2) ± 10 %	1.5
		500 (20) ± 10 %	15

[A] Select the lowest speed that produces rupture in ½ to 5 min for the specimen geometry being used (see 8.2).

[B] See Terminology D 883 for definitions.

[C] The initial rate of straining cannot be calculated exactly for dumbbell-shaped specimens because of extension, both in the reduced section outside the gage length and in the fillets. This initial strain rate can be measured from the initial slope of the tensile strain-versus-time diagram.

9.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods. In cases of disagreement, the tolerances shall be ± 1°C (1.8°F) and ±2 % relative humidity.

NOTE 13—The tensile properties of some plastics change rapidly with small changes in temperature. Since heat may be generated as a result of straining the specimen at high rates, conduct tests without forced cooling to ensure uniformity of test conditions. Measure the temperature in the reduced section of the specimen and record it for materials where self-heating is suspected.

10. Procedure

10.1 Measure the width and thickness of rigid flat specimens (Fig. 1) with a suitable micrometer to the nearest 0.025 mm (0.001 in.) at several points along their narrow sections. Measure the thickness of nonrigid specimens (produced by a Type IV die) in the same manner with the required dial micrometer. Take the width of this specimen as the distance between the cutting edges of the die in the narrow section. Measure the diameter of rod specimens, and the inside and outside diameters of tube specimens, to the nearest 0.025 mm (0.001 in.) at a minimum of two points 90° apart; make these measurements along the groove for specimens so constructed. Use plugs in testing tube specimens, as shown in Fig. 2.

TABLE 2 Modulus, 10^6 psi, for Eight Laboratories, Five Materials

	Mean	S_r	S_R	I_r	I_R
Polypropylene	0.210	0.0089	0.071	0.025	0.201
Cellulose acetate butyrate	0.246	0.0179	0.035	0.051	0.144
Acrylic	0.481	0.0179	0.063	0.051	0.144
Glass-reinforced nylon	1.17	0.0537	0.217	0.152	0.614
Glass-reinforced polyester	1.39	0.0894	0.266	0.253	0.753

10.2 Place the specimen in the grips of the testing machine, taking care to align the long axis of the specimen and the grips with an imaginary line joining the points of attachment of the grips to the machine. The distance between the ends of the gripping surfaces, when using flat specimens, shall be as indicated in Fig. 1. On tube and rod specimens, the location for the grips shall be as shown in Fig. 2 and Fig. 3. Tighten the grips evenly and firmly to the degree necessary to prevent slippage of the specimen during the test, but not to the point where the specimen would be crushed.

10.3 Attach the extension indicator. When modulus is being determined, a Class B-2 or better extensometer is required (see 5.2.1).

NOTE 14—Modulus of materials is determined from the slope of the linear portion of the stress-strain curve. For most plastics, this linear portion is very small, occurs very rapidly, and must be recorded automatically. The change in jaw separation is never to be used for calculating modulus or elongation.

10.3.1 *Poisson's Ratio Determination:*

10.3.1.1 When Poisson's ratio is determined, the speed of testing and the load range at which it is determined shall be the same as those used for modulus of elasticity.

10.3.1.2 Attach the transverse strain measuring device. The transverse strain measuring device must continuously measure the strain simultaneously with the axial strain measuring device.

TABLE 3 Tensile Stress at Yield, 10^3 psi, for Eight Laboratories, Three Materials

	Mean	S_r	S_R	I_r	I_R
Polypropylene	3.63	0.022	0.161	0.062	0.456
Cellulose acetate butyrate	5.01	0.058	0.227	0.164	0.642
Acrylic	10.4	0.067	0.317	0.190	0.897

TABLE 4 Elongation at Yield, %, for Eight Laboratories, Three Materials

	Mean	S_r	S_R	I_r	I_R
Cellulose acetate butyrate	3.65	0.27	0.62	0.76	1.75
Acrylic	4.89	0.21	0.55	0.59	1.56
Polypropylene	8.79	0.45	5.86	1.27	16.5

10.3.1.3 Make simultaneous measurements of load and strain and record the data. The precision of the value of Poisson's ratio will depend on the number of data points of axial and transverse strain taken.

10.4 Set the speed of testing at the proper rate as required in Section 8, and start the machine.

10.5 Record the load-extension curve of the specimen.

10.6 Record the load and extension at the yield point (if one exists) and the load and extension at the moment of rupture.

NOTE 15—If it is desired to measure both modulus and failure properties (yield or break, or both), it may be necessary, in the case of highly extensible materials, to run two independent tests. The high magnification extensometer normally used to determine properties up to the yield point may not be suitable for tests involving high extensibility. If allowed to remain attached to the specimen, the extensometer could be permanently damaged. A broad-range incremental extensometer or hand-rule technique may be needed when such materials are taken to rupture.

11. Calculation

11.1 Toe compensation shall be made in accordance with Annex A1, unless it can be shown that the toe region of the curve is not due to the take-up of slack, seating of the specimen, or other artifact, but rather is an authentic material response.

11.2 *Tensile Strength*—Calculate the tensile strength by dividing the maximum load in newtons (or pounds-force) by the original minimum cross-sectional area of the specimen in square metres (or square inches). Express the result in pascals (or pounds-force per square inch) and report it to three significant figures as tensile strength at yield or tensile strength at break, whichever term is applicable. When a nominal yield or break load less than the maximum is present and applicable, it may be desirable also to calculate, in a similar manner, the corresponding tensile stress at yield or tensile stress at break and report it to three significant figures (see Note A2.8).

11.3 *Percent Elongation*—If the specimen gives a yield load that is larger than the load at break, calculate percent elongation at yield. Otherwise, calculate percent elongation at break. Do this by reading the extension (change in gage length) at the moment the applicable load is reached. Divide that extension by the original gage length and multiply by 100. Report percent elongation at yield or percent elongation at break to two significant figures. When a yield or breaking load less than the maximum is present and of interest, it is desirable to calculate and report both percent elongation at yield and percent elongation at break (see Note A2.2).

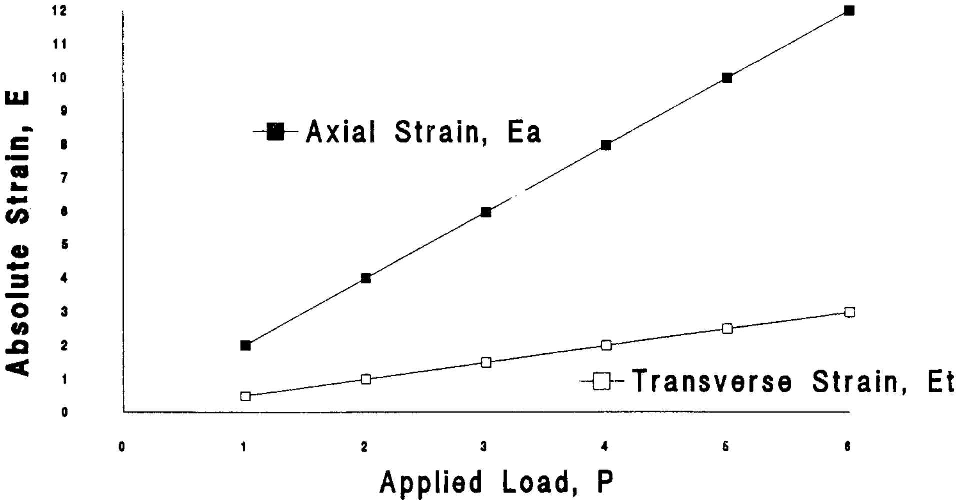


FIG. 4 Plot of Strains Versus Load for Determination of Poisson's Ratio

11.4 *Modulus of Elasticity*—Calculate the modulus of elasticity by extending the initial linear portion of the load-extension curve and dividing the difference in stress corresponding to any segment of section on this straight line by the corresponding difference in strain. All elastic modulus values shall be computed using the average initial cross-sectional area of the test specimens in the calculations. The result shall be expressed in pascals (pounds-force per square inch) and reported to three significant figures.

11.5 *Secant Modulus*—At a designated strain, this shall be calculated by dividing the corresponding stress (nominal) by the designated strain. Elastic modulus values are preferable and shall be calculated whenever possible. However, for materials where no proportionality is evident, the secant value shall be calculated. Draw the tangent as directed in A1.3 and Fig. A1.2, and mark off the designated strain from the yield point where the tangent line goes through zero stress. The stress to be used in the calculation is then determined by dividing the load-extension curve by the original average cross-sectional area of the specimen.

11.6 *Poisson's Ratio*—The axial strain, ϵ_a, indicated by the axial extensometer, and the transverse strain, ϵ, indicated by the transverse extensometers, are plotted against the applied load, P, as shown in Fig. 4. A straight line is drawn through each set of points, and the slopes, $d\epsilon_a / dP$ and $d\epsilon_t / dP$, of these lines are determined. Poisson's ratio, μ, is then calculated as follows:

$$\mu = -(d\epsilon_t / dP)/(d\epsilon_a / dP) \qquad (1)$$

where:

$d\epsilon_t$ = change in transverse strain,
$d\epsilon_a$ = change in axial strain, and
dP = change in applied load;

or

$$\mu = -(d\epsilon_t) / (d\epsilon_a) \qquad (2)$$

11.6.1 The errors that may be introduced by drawing a straight line through the points can be reduced by applying the method of least squares.

11.7 For each series of tests, calculate the arithmetic mean of all values obtained and report it as the "average value" for the particular property in question.

11.8 Calculate the standard deviation (estimated) as follows and report it to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\bar{X}^2) / (n - 1)} \qquad (3)$$

where:

s = estimated standard deviation,
X = value of single observation,
n = number of observations, and
X = arithmetic mean of the set of observations.

11.9 See Annex A1 for information on toe compensation.

TABLE 5 Tensile Strength at Break, 10^3 psi, for Eight Laboratories, Five Materials[A]

	Mean	S_r	S_R	I_r	I_R
Polypropylene	2.97	1.54	1.65	4.37	4.66
Cellulose acetate butyrate	4.82	0.058	0.180	0.164	0.509
Acrylic	9.09	0.452	0.751	1.27	2.13
Glass-reinforced polyester	20.8	0.233	0.437	0.659	1.24
Glass-reinforced nylon	23.6	0.277	0.698	0.784	1.98

[A] Tensile strength and elongation at break values obtained for unreinforced propylene plastics generally are highly variable due to inconsistencies in necking or "drawing" of the center section of the test bar. Since tensile strength and elongation at yield are more reproducible and relate in most cases to the practical usefulness of a molded part, they are generally recommended for specification purposes.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code numbers, form, principal dimensions, previous history, etc.,

12.1.2 Method of preparing test specimens,

12.1.3 Type of test specimen and dimensions,

12.1.4 Conditioning procedure used,

TABLE 6 Elongation at Break, %, for Eight Laboratories, Five Materials[A]

	Mean	S_r	S_R	I_r	I_R
Glass-reinforced polyester	3.68	0.20	2.33	0.570	6.59
Glass-reinforced nylon	3.87	0.10	2.13	0.283	6.03
Acrylic	13.2	2.05	3.65	5.80	10.3
Cellulose acetate butyrate	14.1	1.87	6.62	5.29	18.7
Polypropylene	293.0	50.9	119.0	144.0	337.0

[A] Tensile strength and elongation at break values obtained for unreinforced propylene plastics generally are highly variable due to inconsistencies in necking or "drawing" of the center section of the test bar. Since tensile strength and elongation at yield are more reproducible and relate in most cases to the practical usefulness of a molded part, they are generally recommended for specification purposes.

TABLE 7 Tensile Yield Strength, for Ten Laboratories, Eight Materials

Material	Test Speed, in./min	Values Expressed in psi Units				
		Average	S_r	S_R	r	R
LDPE	20	1544	52.4	64.0	146.6	179.3
LDPE	20	1894	53.1	61.2	148.7	171.3
LLDPE	20	1879	74.2	99.9	207.8	279.7
LLDPE	20	1791	49.2	75.8	137.9	212.3
LLDPE	20	2900	55.5	87.9	155.4	246.1
LLDPE	20	1730	63.9	96.0	178.9	268.7
HDPE	2	4101	196.1	371.9	549.1	1041.3
HDPE	2	3523	175.9	478.0	492.4	1338.5

TABLE 8 Tensile Yield Elongation, for Eight Laboratories, Eight Materials

Material	Test Speed, in./min	Values Expressed in Percent Units				
		Average	S_r	S_R	r	R
LDPE	20	17.0	1.26	3.16	3.52	8.84
LDPE	20	14.6	1.02	2.38	2.86	6.67
LLDPE	20	15.7	1.37	2.85	3.85	7.97
LLDPE	20	16.6	1.59	3.30	4.46	9.24
LLDPE	20	11.7	1.27	2.88	3.56	8.08
LLDPE	20	15.2	1.27	2.59	3.55	7.25
HDPE	2	9.27	1.40	2.84	3.91	7.94
HDPE	2	9.63	1.23	2.75	3.45	7.71

TABLE 9 Tensile Break Strength, for Nine Laboratories, Six Materials

Material	Test Speed, in./min	Values Expressed in psi Units				
		Average	S_r	S_R	r	R
LDPE	20	1592	52.3	74.9	146.4	209.7
LDPE	20	1750	66.6	102.9	186.4	288.1
LLDPE	20	4379	127.1	219.0	355.8	613.3
LLDPE	20	2840	78.6	143.5	220.2	401.8
LLDPE	20	1679	34.3	47.0	95.96	131.6
LLDPE	20	2660	119.1	166.3	333.6	465.6

TABLE 10 Tensile Break Elongation, for Nine Laboratories, Six Materials

Material	Test Speed, in./min	Values Expressed in Percent Units				
		Average	S_r	S_R	r	R
LDPE	20	567	31.5	59.5	88.2	166.6
LDPE	20	569	61.5	89.2	172.3	249.7
LLDPE	20	890	25.7	113.8	71.9	318.7
LLDPE	20	64.4	6.68	11.7	18.7	32.6
LLDPE	20	803	25.7	104.4	71.9	292.5
LLDPE	20	782	41.6	96.7	116.6	270.8

12.1.5 Atmospheric conditions in test room,

12.1.6 Number of specimens tested,

12.1.7 Speed of testing,

12.1.8 Classification of extensometers used. A description of measuring technique and calculations employed instead of a minimum Class-C extensometer system,

12.1.9 Tensile strength at yield or break, average value, and standard deviation,

12.1.10 Tensile stress at yield or break, if applicable, average value, and standard deviation,

12.1.11 Percent elongation at yield or break, or both, as applicable, average value, and standard deviation,

12.1.12 Modulus of elasticity, average value, and standard deviation,

12.1.13 Date of test, and

12.1.14 Revision date of Test Method D 638.

13. Precision and Bias [12]

13.1 *Precision*—Tables 2-6 are based on a round-robin test conducted in 1984, involving five materials tested by eight laboratories using the Type I specimen, all of nominal 0.125-in. thickness. Each test result was based on five individual determinations. Each laboratory obtained two test results for each material.

13.1.1 Tables 7-10 are based on a round-robin test conducted by the polyolefin subcommittee in 1988, involving eight polyethylene materials tested in ten laboratories. For each material, all samples were molded at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of five individual determinations. Each laboratory obtained three test results for each material. Data from some laboratories could not be used for various reasons, and this is noted in each table.

13.1.2 In Tables 2-10, for the materials indicated, and for test results that derived from testing five specimens:

13.1.2.1 S_r is the within-laboratory standard deviation of the average; $I_r = 2.83\ S_r$. (See 13.1.2.3 for application of I_r.)

13.1.2.2 S_R is the between-laboratory standard deviation of the average; $I_R = 2.83\ S_R$. (See 13.1.2.4 for application of I_R.)

13.1.2.3 *Repeatability*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, those test results should be judged not equivalent if they differ by more than the I_r value for that material and condition.

13.1.2.4 *Reproducibility*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, those test results should be judged not equivalent if they differ by more than the I_R value for that material and condition. (This applies between different laboratories or between different equipment within the same laboratory.)

13.1.2.5 Any judgment in accordance with 13.1.2.3 and

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[12] Supporting data are available from ASTM Headquarters. Request RR:D20-1125 for the 1984 round robin and RR:D20-1170 for the 1988 round robin.

13.1.2.4 will have an approximate 95 % (0.95) probability of being correct.

13.1.2.6 Other formulations may give somewhat different results.

13.1.2.7 For further information on the methodology used in this section, see Practice E 691.

13.1.2.8 The precision of this test method is very dependent upon the uniformity of specimen preparation, standard practices for which are covered in other documents.

13.2 *Bias*—There are no recognized standards on which to base an estimate of bias for this test method.

14. Keywords

14.1 modulus of elasticity; percent elongation; plastics; tensile properties; tensile strength

ANNEXES

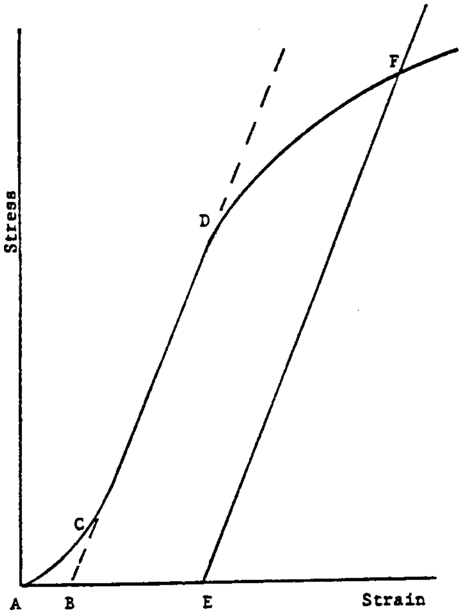
(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, *AC*, that does not represent a property of the material. It is an artifact caused by a takeup of slack and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

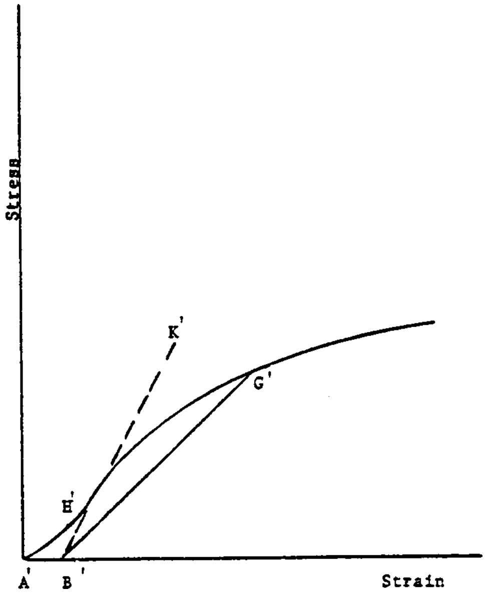
A1.2 In the case of a material exhibiting a region of-Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (*CD*) region of the curve is constructed through the zero-stress axis. This intersection (*B*) is the corrected zero-strain point from which all extensions or strains must be measured, including the yield offset (*BE*), if applicable. The elastic modulus can be determined by dividing the stress at any point along the line *CD* (or its extension) by the strain at the same point (measured from Point *B*, defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (*H*′). This is extended to intersect the strain axis at Point *B*′, the corrected zero-strain point. Using Point *B*′ as zero strain, the stress at any point (*G*′) on the curve can be divided by the strain at that point to obtain a secant modulus (slope of Line *B*′ *G*′). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.



NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region



NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

A2. DEFINITIONS OF TERMS AND SYMBOLS RELATING TO TENSION TESTING OF PLASTICS

A2.1 *elastic limit*—the greatest stress which a material is capable of sustaining without any permanent strain remaining upon complete release of the stress. It is expressed in force per unit area, usually pounds-force per square inch (megapascals).

NOTE A2.1—Measured values of proportional limit and elastic limit vary greatly with the sensitivity and accuracy of the testing equipment, eccentricity of loading, the scale to which the stress-strain diagram is plotted, and other factors. Consequently, these values are usually replaced by yield strength.

A2.2 *elongation*—the increase in length produced in the gage length of the test specimen by a tensile load. It is expressed in units of length, usually inches (millimetres). (Also known as *extension*.)

NOTE A2.2—Elongation and strain values are valid only in cases where uniformity of specimen behavior within the gage length is present. In the case of materials exhibiting necking phenomena, such values are only of qualitative utility after attainment of yield point. This is due to inability to ensure that necking will encompass the entire length between the gage marks prior to specimen failure.

A2.3 *gage length*—the original length of that portion of the specimen over which strain or change in length is determined.

A2.4 *modulus of elasticity*—the ratio of stress (nominal) to corresponding strain below the proportional limit of a material. It is expressed in force per unit area, usually megapascals (pounds-force per square inch). (Also known as *elastic modulus* or *Young's modulus*).

NOTE A2.3—The stress-strain relations of many plastics do not conform to Hooke's law throughout the elastic range but deviate therefrom even at stresses well below the elastic limit. For such materials the slope of the tangent to the stress-strain curve at a low stress is usually taken as the modulus of elasticity. Since the existence of a true proportional limit in plastics is debatable, the propriety of applying the term "modulus of elasticity" to describe the stiffness or rigidity of a plastic has been seriously questioned. The exact stress-strain characteristics of plastic materials are very dependent on such factors as rate of stressing, temperature, previous specimen history, etc. However, such a value is useful if its arbitrary nature and dependence on time, temperature, and other factors are realized.

A2.5 *necking*—the localized reduction in cross section which may occur in a material under tensile stress.

A2.6 *offset yield strength*—the stress at which the strain exceeds by a specified amount (the offset) an extension of the initial proportional portion of the stress-strain curve. It is expressed in force per unit area, usually megapascals (pounds-force per square inch).

NOTE A2.4—This measurement is useful for materials whose stress-strain curve in the yield range is of gradual curvature. The offset yield strength can be derived from a stress-strain curve as follows (Fig. A2.1):

On the strain axis lay off *OM* equal to the specified offset.

Draw *OA* tangent to the initial straight-line portion of the stress-strain curve.

Through *M* draw a line *MN* parallel to *OA* and locate the intersection of *MN* with the stress-strain curve.

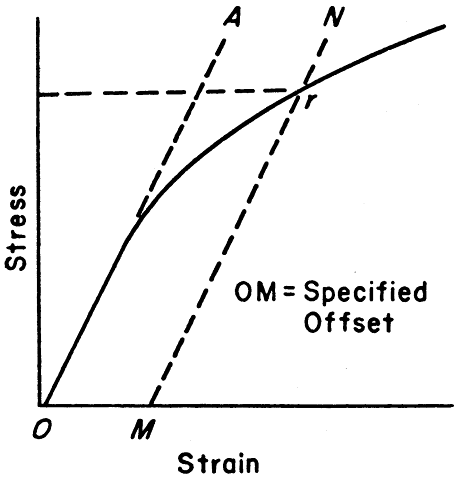


FIG. A2.1 Offset Yield Strength

The stress at the point of intersection *r* is the "offset yield strength." The specified value of the offset must be stated as a percent of the original gage length in conjunction with the strength value. *Example:* 0.1 % offset yield strength = ... MPa (psi), or yield strength at 0.1 % offset ... MPa (psi).

A2.7 *percent elongation*—the elongation of a test specimen expressed as a percent of the gage length.

A2.8 *percent elongation at break and yield:*

A2.8.1 *percent elongation at break*

the percent elongation at the moment of rupture of the test specimen.

A2.8.2 *percent elongation at yield*

the percent elongation at the moment the yield point (A2.21) is attained in the test specimen.

A2.9 *percent reduction of area (nominal)*—the difference between the original cross-sectional area measured at the point of rupture after breaking and after all retraction has ceased, expressed as a percent of the original area.

A2.10 *percent reduction of area (true)*—the difference between the original cross-sectional area of the test specimen and the minimum cross-sectional area within the gage boundaries prevailing at the moment of rupture, expressed as a percentage of the original area.

A2.11 *proportional limit*—the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law). It is expressed in force per unit area, usually megapascals (pounds-force per square inch).

A2.12 *rate of loading*—the change in tensile load carried by the specimen per unit time. It is expressed in force per unit time, usually newtons (pounds-force) per minute. The initial rate of loading can be calculated from the initial slope of the load versus time diagram.

A2.13 *rate of straining*—the change in tensile strain per unit time. It is expressed either as strain per unit time, usually metres per metre (inches per inch) per minute, or percent elongation per unit time, usually percent elongation per minute. The initial rate of straining can be calculated from the initial slope of the tensile strain versus time diagram.

NOTE A2.5—The initial rate of straining is synonymous with the rate of crosshead movement divided by the initial distance between crossheads only in a machine with constant rate of crosshead movement and when the specimen has a uniform original cross section, does not "neck down," and does not slip in the jaws.

A2.14 *rate of stressing (nominal)*—the change in tensile stress (nominal) per unit time. It is expressed in force per unit area per unit time, usually megapascals (pounds-force per square inch) per minute. The initial rate of stressing can be calculated from the initial slope of the tensile stress (nominal) versus time diagram.

NOTE A2.6—The initial rate of stressing as determined in this manner has only limited physical significance. It does, however, roughly describe the average rate at which the initial stress (nominal) carried by the test specimen is applied. It is affected by the elasticity and flow characteristics of the materials being tested. At the yield point, the rate of stressing (true) may continue to have a positive value if the cross-sectional area is decreasing.

A2.15 *secant modulus*—the ratio of stress (nominal) to corresponding strain at any specified point on the stress-strain curve. It is expressed in force per unit area, usually megapascals (pounds-force per square inch), and reported together with the specified stress or strain.

NOTE A2.7—This measurement is usually employed in place of modulus of elasticity in the case of materials whose stress-strain diagram does not demonstrate proportionality of stress to strain.

A2.16 *strain*—the ratio of the elongation to the gage length of the test specimen, that is, the change in length per unit of original length. It is expressed as a dimensionless ratio.

A2.17 *tensile strength (nominal)*—the maximum tensile stress (nominal) sustained by the specimen during a tension test. When the maximum stress occurs at the yield point (A2.21), it shall be designated tensile strength at yield. When the maximum stress occurs at break, it shall be designated tensile strength at break.

A2.18 *tensile stress (nominal)*—the tensile load per unit area of minimum original cross section, within the gage boundaries, carried by the test specimen at any given moment. It is expressed in force per unit area, usually megapascals (pounds-force per square inch).

NOTE A2.8—The expression of tensile properties in terms of the minimum original cross section is almost universally used in practice. In the case of materials exhibiting high extensibility or necking, or both (A2.15), nominal stress calculations may not be meaningful beyond the yield point (A2.21) due to the extensive reduction in cross-sectional area that ensues. Under some circumstances it may be desirable to express the tensile properties per unit of minimum prevailing cross section. These properties are called true tensile properties (that is, true tensile stress, etc.).

A2.19 *tensile stress-strain curve*—a diagram in which values of tensile stress are plotted as ordinates against corresponding values of tensile strain as abscissas.

A2.20 *true strain* (see Fig. A2.2) is defined by the following equation for ϵ_T:

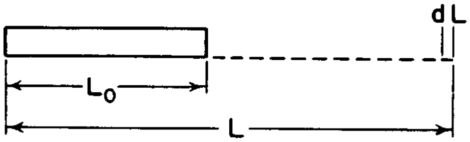


FIG. A2.2 Illustration of True Strain Equation

$$\epsilon_T = \int_{L_o}^{L} dL/L = \ln L/L_o \qquad \text{(A2.1)}$$

where:

dL = increment of elongation when the distance between the gage marks is L,
L_o = original distance between gage marks, and
L = distance between gage marks at any time.

A2.21 *yield point*—the first point on the stress-strain curve at which an increase in strain occurs without an increase in stress (Fig. A2.2).

NOTE A2.9—Only materials whose stress-strain curves exhibit a point of zero slope may be considered as having a yield point.

NOTE A2.10—Some materials exhibit a distinct "break" or discontinuity in the stress-strain curve in the elastic region. This break is not a yield point by definition. However, this point may prove useful for material characterization in some cases.

A2.22 *yield strength*—the stress at which a material exhibits a specified limiting deviation from the proportionality of stress to strain. Unless otherwise specified, this stress will be the stress at the yield point and when expressed in relation to the tensile strength shall be designated either tensile strength at yield or tensile stress at yield as required in A2.17 (Fig. A2.3). (See *offset yield strength.*)

A2.23 *Symbols*—The following symbols may be used for the above terms:

Symbol	Term
W	Load
ΔW	Increment of load
L	Distance between gage marks at any time
L_o	Original distance between gage marks
L_u	Distance between gage marks at moment of rupture
ΔL	Increment of distance between gage marks = elongation
A	Minimum cross-sectional area at any time
A_o	Original cross-sectional area
ΔA	Increment of cross-sectional area
A_u	Cross-sectional area at point of rupture measured after breaking specimen
A_T	Cross-sectional area at point of rupture, measured at the moment of rupture
t	Time
Δt	Increment of time
σ	Tensile stress
$\Delta\sigma$	Increment of stress
σ_T	True tensile stress
σ_U	Tensile strength at break (nominal)
σ_{UT}	Tensile strength at break (true)
ϵ	Strain
$\Delta\epsilon$	Increment of strain
ϵ_U	Total strain, at break
ϵ_T	True strain
$\%El$	Percentage elongation
Y.P.	Yield point
E	Modulus of elasticity

A2.24 Relations between these various terms may be defined as follows:

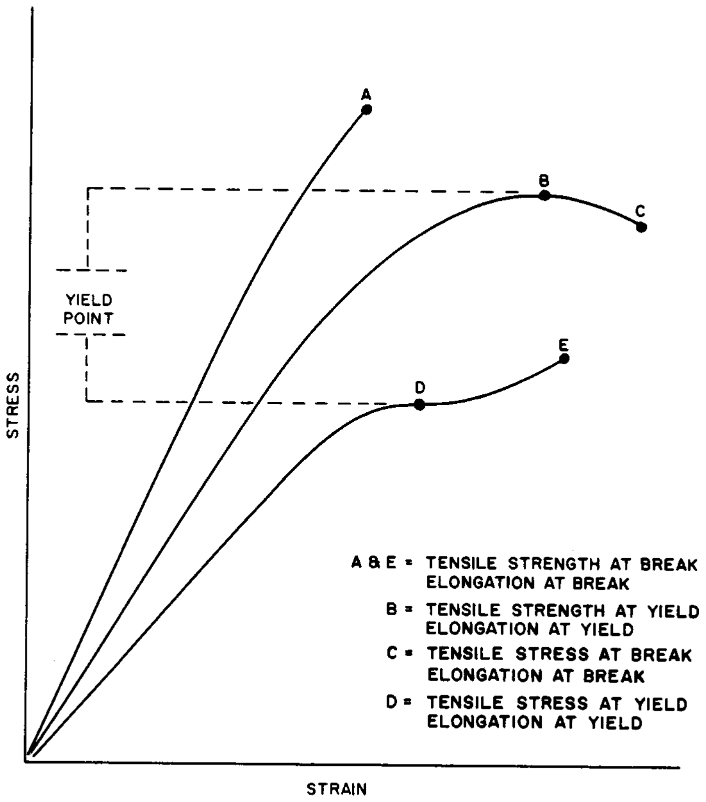


FIG. A2.3 Tensile Designations

σ = W/A_o
σ_T = W/A
σ_U = W/A_o (where W is breaking load)
σ_{UT} = W/A_T (where W is breaking load)
ϵ = $\Delta L/L_o = (L - L_o)/L_o$
ϵ_U = $(L_u - L_o)/L_o$
ϵ_T = $\int_{L_o}^{L} dL/L = \ln L/L_o$
$\%El$ = $[(L - L_o)/L_o] \times 100 = \epsilon \times 100$

Percent reduction of area (nominal) = $[(A_o - A_u)/A_o] \times 100$
Percent reduction of area (true) = $[(A_o - A_T)/A_o] \times 100$
Rate of loading = $\Delta W/\Delta t$
Rate of stressing (nominal) = $\Delta\sigma/\Delta = (\Delta W]/A_o)/\Delta t$
Rate of straining = $\Delta\epsilon/\Delta t = (\Delta L/L_o)\Delta t$

For the case where the volume of the test specimen does not change during the test, the following three relations hold:

$$\sigma_T = \sigma(1 + \epsilon) = \sigma L/L_o \qquad \text{(A2.2)}$$

$$\sigma_{UT} = \sigma_U(1 + \epsilon_U) = \sigma_U L_u/L_o$$

$$A = A_o/(1 + \epsilon)$$

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 638–98:

(1) Revised 10.3 and added 12.1.8 to clarify extensometer usage.

(2) Added 12.1.14.

(3) Replaced reference to Test Methods D 374 with Test Method D 5947 in 2.1 and 5.3.

D 638–99:

(1) Added and clarified extensometer classification requirements.

D 638–00:

(1) Added 11.1 and renumbered subsequent sections.

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