EFFECT OF CARBON NANOTUBE ON THE PROPERTIES OF GRAPHITE - CARBON BLACK-EPOXY COMPOSITE FOR BIPOLAR PLATE

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Bachelor of Mechanical Engineering (Structure & Materials)"

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This report is submitted to Faculty of Mechanical Engineering as a requirement to get award of Degree of Mechanical Engineering (Structure & Material)

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DECLARATION

"I here declare that the work in this report is my own expect for summaries and quotations which have been duly acknowledgment"

Signature :
Author :
Date :

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ABSTRACT

The conductive polymer composite(CPCs) bipolar plate had been the target to replace the pure-graphite. This is due to the low manufacturing cost and light of total mass of bipolar plate in PEMFCs. The aim of this research is to study the effect of CNTs on the properties of Gr/CB/EP composite. The second aim of this research is to determine the critical loading of CNTs in Gr/CB/EP composite. The ratio of fillers (Gr/CNT) and binder (EP) is fixed at 75:25. The amount of CB also will be fixed 25% and by adding the small amount of CNTs in to Gr/CNT/EP composite thus will gives synergy effects on electrical conductivity and mechanical properties. The small amount of CNTs which is 0, 5, 10 and 15 wt.% (from the total weight of fillers 75%) will be added into Gr/CB/EP composite. Before the fabrication process using the hot press, the filler of Gr/CB/CNT will be mixed used ball mill. In order the determines the effect of CNTs content in Gr/CNT/EP composite, the test such as electrical conductivity, flexure test, density test hardness and microstructure analysis has been carried out. The best weight of CNTs contents are at 10wt% in Gr/CB/EP.

ABSTRAK

Polimer konduksian plat dwikutub komposit (CPCs) telah menjadi sasaran menggantikan grafit tulen. Ini adalah disebabkan kos perkilangan rendah.Tujuan penyelidikan ini adalah untuk belajar kesan CNTs pada Gr/CB/EP.Tujuan kedua penyelidikan ini adalah untuk menentukan pemuatan kritikal CNTs dalam Gr/CB/EP. Nisbah pengisi (Gr/CNT) dan pengikat (EP) ditetapkan pada 75:25. Jumlah CB juga akan ditetapkan 25% dan dengan menambahkan sedikit CNTs dalam kepada Gr/CNT/EP maka akan memberi kesan-kesan sinergi di kekonduksian elektrik dan sifat mekanik. Sedikit CNTs yang merupakan 0, 5, 10 dan 15 berat.% (dari jumlah berat pengisi 75%) akan ditambah ke dalam Gr/CB/EP. Sebelum proses pembuatan menggunakan penekan panas, pengisi Gr / CB / CNT akan bercampur dengan mengunakan pengisar bola.Dalam usaha menentukan kesan CNTs dalam Gr / CNT / rencam EP, ujian seperti kekonduksian elektrik, ujian kekuatan lenturan, kekerasan ujian kepadatan dan analisis mikrostruktur telah dilaksanakan. Berat terbaik kandungan CNTs berada di 10wt% dalam Gr / CB / EP.

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LIST OF ABBEREVATIONS

CNTs	Carbon Nanotubes
CB	Carbon Black
Gr	Graphite
EP	Epoxy
PEMFCs	Polymer Electrolyte Membrane Fuel Cell
DC	Direct Current
DOE	Department of Environment
CPC	Conductive Polymer Compositions

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Fuel cell in today plays an important role for the progress of a country. Therefore, to reduce energy consumption a lot, have focused on environmental factors. Therefore, Proton Exchanges Membrane Fuel Cells (PEMFCs) are introduced. So, at low temperatures PEMFCs will converts hydrogen and oxygen into electrical energy. In terms of transportation, PEMFC fuel cell is being developed. It is a fuel cell that does not move and the portable fuel cell. PEMFCs is also known as Polymer Electrolyte Membrane (PEM).

Bipolar plate (BPs) has become a very hot issue of research and critical. In research carried out, BPs must have a specifications, application, electrical and also thermal conductivity. The application must present physical properties and chemistry. Therefore, combination of reinforce of BPs material has been used to archived good electrical conductivity. The Department of Environment (DOE) has set the characteristics of the ideal Bipolar Plates. The fabrication of BPs should be satisfied. The properties requirement shown in Table 1.1.

Table 1.1 Properties specification by DOE (source : Yeetsorn et al, 2012)

Properties	**Value**
Electrical conductivity	>100
Thermal conductivity	>10
Flexural strength	>25[MPa]
Shore Hardness	>50
Bulk Density	<5[g/c]

1.2 PROBLEM STATEMENT

Nowadays, Polymer Electrolyte Membrane Fuel Cell (PEMFC) get more researches. Due to they want to change of pure graphite or metal based bipolar plates. This is because it more attractive of high power density, low operating temperature and converting fuel to water. Based on a study did by Selamat M.Z (2013) is the most commonly used of bipolar plate materials are graphite materials. Due to the graphite have excellent corrosion resistance, high electrical conductivity, and a lower density than the other materials. But, the disadvantages graphite materials is very high cost of machining channels into the surface and their vulnerability will cause the fuel cell stack becomes heavy and voluminous. Conducting Polymer Composite (CPC) is fabricate from the mixed of conductive fillers such as Gr/CB/CNT had been incorporated in EP as matrix for fabrication of electrical conductive polymer composite plate. The advantages of CPC are easy of shaping, low density, and wide range of electrical conductivities as well as corrosion resistance.

1.3 OBJECTIVE

The main objective of this research is:

1. To study the effect of Carbon Nanotubes (CNT) on the properties of Graphite(Gr), Carbon Black(CB), and Epoxy (EP) composite.
2. To determine the critical loading of CNT in Gr, CB, and EP composite.

1.4 SCOPE OF PROJECT

This research will study the effect of carbon Nanotubes (CNT) loading on the electrical and mechanical properties of Gr, CNT, and EP composite. The ratio of fillers (Gr/CNT) and binder (EP) is fixed at 75:25. The amount of CB also will be fixed 25% and by adding the small amount of CNTs in to Gr/CNT/EP composite thus will gives synergy effects on electrical conductivity and mechanical properties, The small amount of CNTs which is 0, 5, 10 and 15 wt.% (from the total weight of fillers 75%) will be added into Gr/CB/EP composite. Before the fabrication process using the hot press, the filler of Gr/CB/CNT will be mixed used ball mill. In order the determines the effect of CNTs content in Gr/CNT/EP composite, the test such as electrical conductivity, flexure test, density test hardness and microstructure analysis will be performed.

CHAPTER 2

LITERATURE REVIEW

2.1 FUEL CELL

Fuel cells is a device of electrochemically which changes the chemical from energy of gaseous or liquid reactants into direct current (DC) electricity. In the simplest case of fuel cell, it was operating with hydrogen (fuel) and oxygen (air) as reacting gases. Figure 2.1 shows the basic of operation fuel cell. A proton or oxide ion was current equivalent to the electrolyte and parts of the homogeneous electrodes structure.

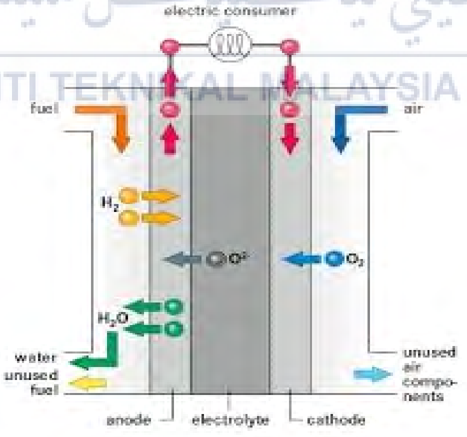


Figure 2.1 : Basic Fuel Cell
(fuel cell & hydrogen energy Association 2015)

All types of fuel cells require a fuel that reacts with oxygen and in the process releases the chemical energy contained in the fuel. However, the energy is not discharged in the form of heat but as electrical energy.

The electrons released in this process are transported by an external electric circuit to the load, thus providing electric energy. Compared to conventional power plants in which heat from the combustion of fuels is converted into electricity mechanically (by means of generators), in fuel cells, a substantial part of the energy losses involved in the power plant process is avoided because there is no combustion step.

Although in principle, fuel cells have a very simple structure, it is a challenge to identify materials that have the required properties, and are able to convert fuel and oxygen into usable electricity. Even when suitable materials have been found, another difficulty consists of giving them the right shape and making sure that the device built from these materials are robust and have a long lifetime.

2.2 POLYMER ELECTROLYTE MEMBRANCE FUEL CELL (PEMFCs)

The purpose of Proton-exchange membrane fuel cells (PEMFCs) are to changes the energy for suitable applications with difference requirements. The material combinations of PEMFCs that used also must difference. But need to get the properties of bipolar plates the properties for bipolar plates are as follows:

- Have electrical conductivity that >100 S cm bulk conductivity.
- The thermal conductivity must >20Wcm
- High chemical and corrosion resistance.
- Mechanical stability toward compression forces.
- The low permeability for hydrogen.
- Mass production techniques must have low-cost material being process able.
- Low volume and weight.

2.3 MAIN COMPONENT OF FUEL CELL

Figure 2.2 shows the structure diagram of PEMFCs. There are three main components of PEMFCs which are Membrane Electrode Assembly (MEA) Bipolar plate (BPs) and End plate. The MEA is the heart component of PEMFCs and work currently being done to find cheaper and thinner membranes whilst maintaining durability.

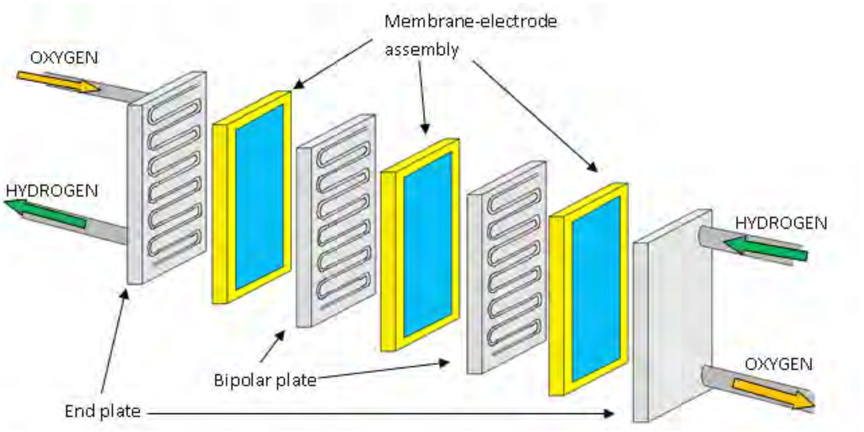


Figure 2. : Structure Diagram PEMFCs.
(Source : Yilser Devri,2012)

2.4 CONDUCTIVE POLYMER COMPOSITES(CPCs)

Conductive polymer composites (CPC) and coated-metallic bipolar plates are widely studied as alternative materials to conventional carbon-carbon plates. Polymer based BPs use thermoplastics, thermosets or elastomers as matrix material and to ensure high electrical conductivities. That contain carbon-based conductive fillers such as Gr, CB and CNTs. These composites are produced via melt mixing and injection or compression moulding. They possess good corrosive behaviour due to inert structure of polymer.

Moulding techniques shorten the production cycle-time, carry out porous-free bipolar plates and facilitate flow field design. As polymer itself behaves like insulating material, to convert it into conductive phase requires attentive introduction of conductive fillers into the matrix. The higher the concentration of filler, the higher the probabilities have to a porous structure or inferior mechanical properties. The dispersion state of filler is another important issue to take care. Especially high surface area fillers such as carbon nanotubes tend to create agglomerates due to inter-particle Van der Waals forces. To benefit from their properties, those agglomerates should be well disentangled and homogeneous filler dispersion should be maintained

2.5 MATERIALS

The materials used to produce CPCs samples are Gr, CB and CNTs powder. The binder in this research is Epoxy. Table 2.1 shows the main materials properties

Table 2.1. : Material Properties of Gr/CB/CNTs/EP

(Source : Selamat,et al.2013)

Material	Gr	CB	CNTs	EP
Grade	3243	5303	NC 7000	105/206
Density	1.74 g/cm^3	1.7-1.9 g/cm^3	1.0 g/cm^3	1.15 g/cm^3
Thermal stability	3500-4000 ^{0}C	3000 ^{0}C	>700 ^{0}C	180-220 ^{0}C
Size	$\leq 60\mu m$	$\leq 5\mu m$	9.5 nm (diameter) 1.5 μm (length)	-
Resistivity	0.036 Ωcm	0.314 Ωcm	5-5 $\mu\Omega cm$	1(1014 Ωm)

2.5.1 Fillers

In order to achieve a good electrical conductivity of the composite, the combination of multi-fillers have been used as bipolar plate. The reinforcement fillers used commonly used including Gr, CB, and CNTs. Those fillers are which have been incorporated into the composite.

2.5.1.1 Carbon Black (CB)

Black carbon is carbon that is controlled in the production process and forms aggregates also vary in size, porosity and surface chemistry. Carbon Black typically contains more than 95 % pure carbon with minimal quantities of oxygen, hydrogen and nitrogen. In the manufacturing process that uses carbon black, carbon black particles will form different shapes. The size of the carbon black is 10nm to 500nm. The combine into a chain-like aggregates, which determines the structure of individual carbon black grades.

While in the polymer industry, to obtain black fine particles of carbon black used. The main function of carbon black is the ability to absorb UV light in turn convert it to produced heat. Thus, the manufacture of polymers such as polypropylene and polyethylene are becoming more resistant and can prevent contamination by UV radiation from sunlight. In addition, carbon black is used as the insulating polymer in the production of wire and cable. It also can improve the insulating properties of polystyrene, which is widely used in construction

Figure 2.3: Carbon Black

2.5.1.2 Graphite (Gr)

Graphite is the most crystalline form of carbon, apart from diamond and fullerenes. Figure below shown the graphite. It exhibits the properties both metal such as thermal and electrical conductivity and of a non-metal such as inertness, high thermal resistance and lubricity. Based on the research of Saparuddin,(2012) it stated that optimization of the property is only possible if the percolation threshold of the conductive filler in the polymer matrix is known. Polymer based composites BPs achieve their electrical performance by the incorporation of specific loading of conductive inorganic.

Figure 2.4: Graphite

2.5.1.3 Carbon Nanotube (CNTs)

Nanoscopic particles of carbon nanotubes are tubular structures formed by carbon atoms. Diameter size between 1 and 50 nm for the normal duration of one micrometre up to a few centimetres. Therefore, the ratio of CNTs can be very large. Advantages of CNTs are available in commercial form multiwall or in the laboratory as a wall. Since their discovery, the physical characteristics unique has led to enormous interest. With a very large elastic modulus, carbon nanotubes known as a reinforcing agent effectively. Depending on their molecular structure, carbon nanotubes with small diameter show either semi-conducting or metallic behaviour.

Figure 2.5: Carbon Nanotube (CNTs)

2.6 MANUFACTURING PROCESS

There are some manufacturing method that can be used to fabricate bipolar plate. Compression moulding and injection moulding are two main processes for current manufacturing of bipolar plates.

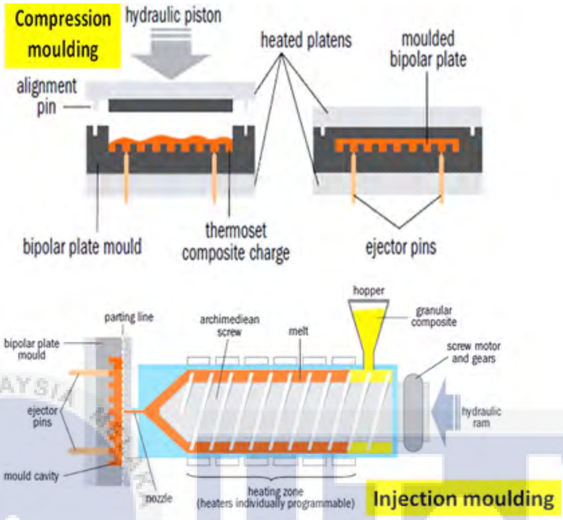


Figure 2.6: Manufacturing Method (Source : Yeetsorn, et al.2011)

2.6.1 Injection Mould

Injection moulding machines is thought of as an attractive manufacturing method for composite bipolar plates. This is due to the high productivity inherent to the process that need to favour the massive production rates necessary to reduce production cost. Furthermore, by using the injection moulding is a low cost and high productivity manufacturing method.

However, other process like extrusion with appropriated die, rolling and thermoforming may be alternative progressions to producing composited bipolar plate. Although the injection moulding process favours the massive production rates that are essential to reduce production cost, the electrical conductivity. At the same time, the high shearing will promotes the rupture of conductive network structure in bipolar plate composition.

2.6.2 Compression Mould

The compression moulding is a very commonly method for mass production of polymer composites bipolar plate. Compression moulding is stared with the powder compound or bend prepared in an extruder or an internal mixer at a proper temperature from a polymer binder resin and conductivity filler particle. The compression moulding is favoured for both thermoplastic and thermosetting matric composites. Even though both are favoured by compression moulding, once if compression moulding is used, the thermoset has to be cured, and while thermoplastic material has to be cooled. Production time for both process are sharply increase.

Furthermore, the cost-effective mass production would be more readily achievable with thermosets rather than thermoplastics it is due to the shorter cycle times for thermosets. In addition by using compression method the compression moulded thermoset composite can be cured comfortably in less than 10 minutes, resulting in cycle times less than those required for thermoplastics.

2.7 TESTING METHOD

Some testing methods have to do on bipolar plate to determine the properties of the sample. The commonly test such as electrical and mechanical properties.

2.7.1 Electrical Conductivity

Electrical conductivity is the measure of the amount of electrical current a material can carry or it's ability to carry a current. Electrical conductivity is also known as specific conductance. Conductivity is an intrinsic property of a material.

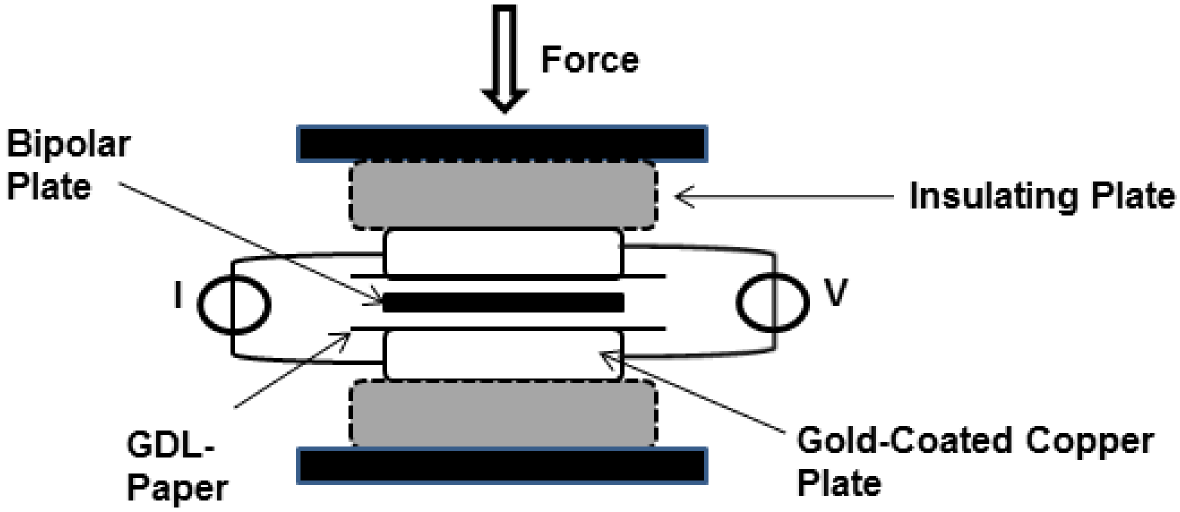


Figure 2.7 Through-plane electrical conductivity setup. (Source:Rungsima Yeetsorn, 2011)

2.7.2 Mechanical Properties

In this research, the mechanical properties common properties need to test such as flexural strength, bulk density test and hardness test. The flexural strength of the composite bipolar plates are important as the bipolar plates may undergo bending force during clamping with the fuel cell hardware. For bulk density measurement, density of the composite bipolar plate was determined by dry density measurement method. Next, the hardness will be measured with the help of stereoscopic hardness tester.

CHAPTER 3

METHODOLOGY

3.1 EXPERIMENTAL OVERVIEW

This section will present the methodology that will apply in the research. Figure 3.1 shows the flow chart of methodology process for Gr, CB, CNTs and EP composite for BPs in PEMFCs.

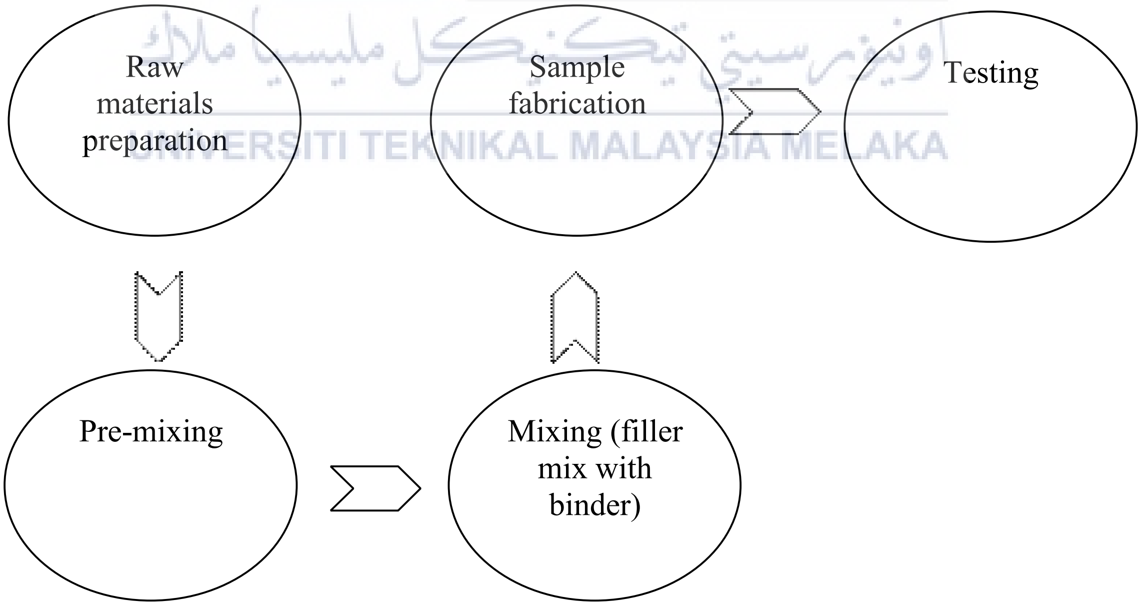


Figure 3.1: Flow chart of methodology process

3.2 RAW MATERIALS

In this study, the materials selected for composite development includes polymer matrix and conductive fillers. Therefore, the chosen materials that need in this research are Gr, CB, and CNTs as filler conductive. Meanwhile, EP was chosen to be a polymer matrix as a binder of bipolar plate for this research. Those materials were chosen instead of metals is due to corrosion resistance and less cost during fabrication.

3.3 FABRICATION METHOD

There are common type for fabrication method which is compression method. Before applying with this fabrication method, some steps need to be carry on like preparing raw materials according with its characterization then pre-mixing for those filler by ball mill machine and follow by using internal mixer machine to mix with the polymer. After this, compression moulding will be taking over and a specimen of BPs will be form up.

3.3.1 Characterization of Raw Materials

In this research, the specific conductive fillers which are Gr, CB and CNTs ratio and weight percentage of EP is generalized. The ratio of fillers (Gr, CB and CNTs) and binder (EP) is fixed at 75:25. The adding of small amount of CNTs in to Gr, CB and EP composite thus will gives synergy effect on electrical conductivity and mechanical properties. In this research of the experiment, three sample with differences composition of Gr, CB and CNTs and EP and also differences temperature were selected in Table 3.1 with its weight percent.

Table 3.1: The composition of composite Gr/CB/CNT/EP

Gr	**CB**	**CNT**	**EP**	**Temperature (C^0)**
45	25	5	25	80
40	25	10	25	
35	25	15	25	
45	25	5	25	90
40	25	10	25	
35	25	15	25	
45	25	5	25	100
40	25	10	25	
35	25	15	25	

3.3.2 Pre-Mixing

A process of pre mixing for polymer Nano composite is using ball mill. The possibility of using ball mill to produce nano scale reinforcement has been verified instead of trying to discover low cost processes. In this research will study the effectiveness of conductivity of CNTs. Therefore, all materials as The composition of composite Gr,CB,and CNTs composites are mixed using ball mill for mixing process.

Figure 3.2: Ball Mill

The materials were weighted based on the composition that has been specified as shown in Table 3.2. Therefore, a container with fill some of steel balls was filled with raw material in powder form which were weighted. The materials are poured slowly because those materials are too light. The machined was run with the time rate around one and half hours to ensure that the combination of fillers is well-mixed.

Table 3.2 : Composition of CNTs, Gr and CB

CNTs		**Gr**		**CB**	
%	**gram**	**%**	**gram**	**%**	**gram**
5	6.67	45	60	25	33.33
10	13.33	40	53.33	25	33.33
15	20.00	35	46.67	25	33.33

3.3.3 Internal Mixing Process

In this process, Gr/CB/CNTs will be mix with EP by using lab used blender for internal mixing process among filler and binder. The ratio of each composition is shown in Table 3.2. The mechanism of stirrer machines is about cylindrical containers in which the materials are deformed by rotating blades or rotor. These machines are used in the compounding of the raw material.

Figure 3.3: Blender

3.3.4 Compression Moulding

Compression moulding method has been chosen for a compress part for this experiment. This process is using a Hot Press Machines. The composites are carefully and immediately filled into the mould.

Figure 3. 4: Hot Press Machines.

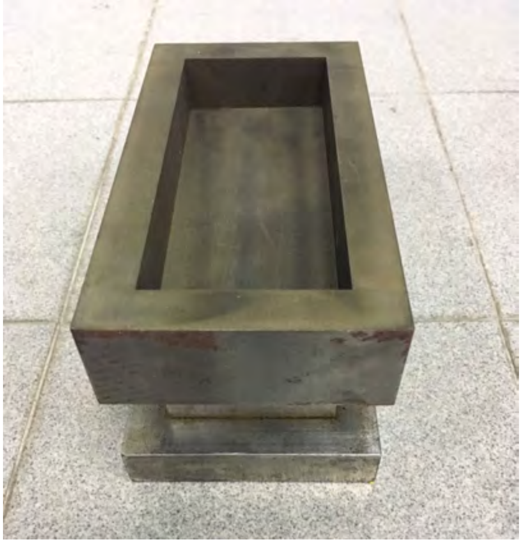


Figure 3.5: Moulding

3.4 TESTING METHOD

This method will carry out the properties of the bipolar plate. There are included electrical conductivity, bulk density, shore hardness and flexure strength.

3.4.1 Electrical Conductivity

The electrical conductivity of the sample was measured using Jandel Multi Four Point Probe technique at a constant current supply. In this method, outer probes apply current to the sample and the inner two probe measure the voltages

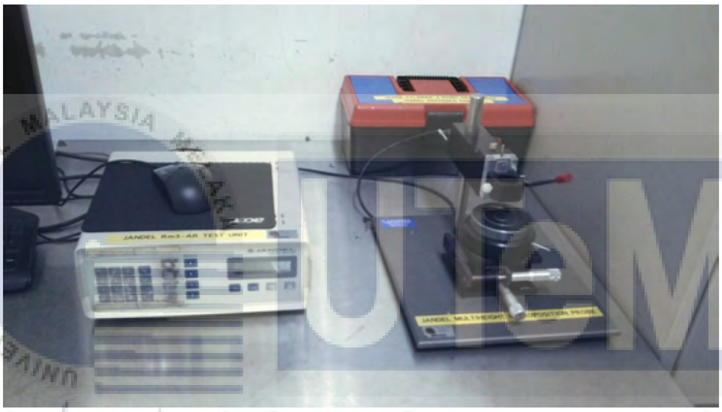


Figure 3.6: Jandel Multi.

3.4.2 Flexural Strength

Flexural strength was measured by the three point method using the Instron Universal Testing Machine.

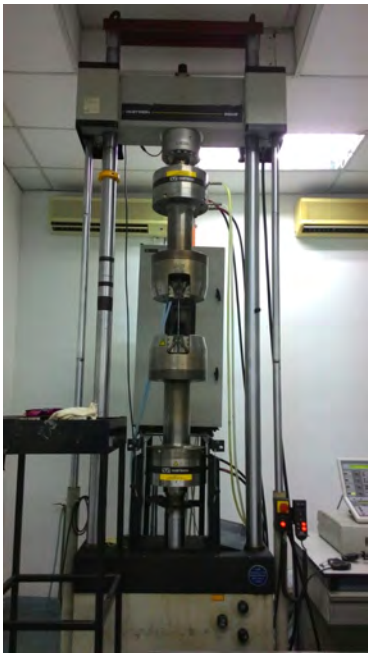


Figure 3.7: Instron Universal Testing Machine

3.4.3 Bulk Density

The composite bipolar plate were cut with a Proxxon Table Saw to dimension to measure the bulk density by means of the well-known dry bulk density measurement method according to ASTM C559-10 by using Electronic Densimeter.

Figure3.8: Electronic Densimeter.

3.4.4 Shore Hardness

Shore hardness was measured with the help of stereoscopic tester. As show below by Figure 3.9 .The hardness of the composites bipolar plate was measured that involved dropping a diamond tipped hammer, which falls inside a composite plate under the force of its own weight from a fixed height, onto the test specimen.

Figure 3.9: Shore Hardness

3.4.5 Scanning Electronic Microscopic

Scanning Electron Microscopy. A scanning electron microscopic (SEM) scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition.

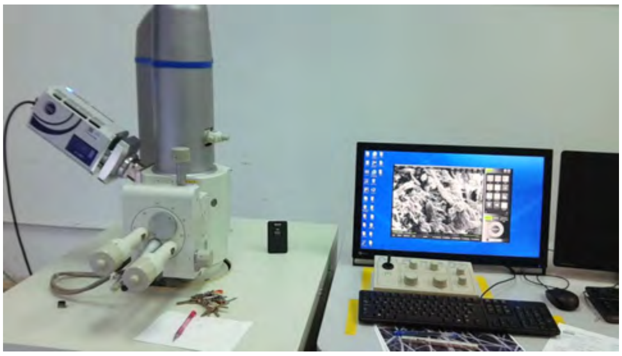


Figure 3.10 :Scanning Electrone microscopic

CHAPTER 4

RESULTS AND ANALYSIS

4.1 FINAL COMPOSITION

In this research, the conductive fillers which are Gr, CB , and CNTs ratio and weight percentage of the EP is generalized. The ratio of filler (Gr/ CB/CNTs) and binder (EP) is fixed at 75 : 25. By adding the CNTs in this composition and with difference temperature it will give difference effect and result based on DOE target. After done the experiment, some of weight had been changed due to fail of mixing process. Figure 4.1 shown the sample of composition bipolar plate after mixing process.

Figure 4.1 Sample of Gr/ CB/ CNTs

4.2 SAMPLE FABRICATION

By using Hot Press Machines, we done compression moulding to get the sample. When carrying out this process, some of parameters had been changes due to the failed during the experiment. Figure 4.2 showed the sample with difference composition ratio of materials and difference temperature. While Table 4.1 showed the parameters that applied on this experiment.

Figure 4.2 Sample of Gr/ CB/ CNTs after Compress Moulding

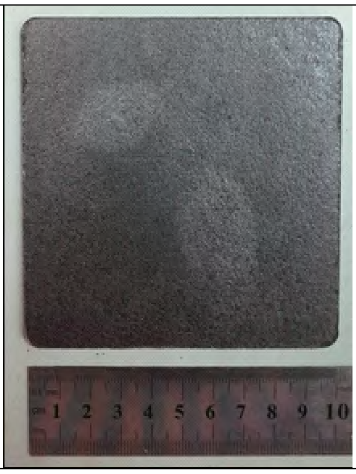
Table 4.1: Parameter of Specimen

Sample	Composite mixing time (min)	Pressure (tones)	Temperature ($°c$)	Pre-Heat (minute)	Compressing (min)
1	5	60	80	6	30
2	5	60	80	6	30
3	5	60	80	6	30
4	5	60	90	6	30
5	5	60	90	6	30
6	5	60	90	6	30
7	5	60	100	6	30
8	5	60	100	6	30
9	5	60	100	6	30

4.3 TESTING RESULT

4.3.1 NET EYE OBSERVATION

Table 4.2: Sample of Gr/ CB/CNT/EP

ºc / CNT	80	90	100
5			
10			
15			

4.3.2 ELECTRICAL CONDUCTIVITY RESULT

The electrical conductivity of samples was tested by using Jandel Multi Four Probe Technique at constant current supply 1 mA. Figure 4.3 showed the location point of data reading.

Figure 4.3: Location data reading of electrical conductivity testing

Table 4.3: Electrical Conductivity of each sample

°C **CNT**	**80**	**90**	**100**
5	77.83	126.23	117.64
10	161.18	74.1	226.7
15	196.99	141.32	123.92

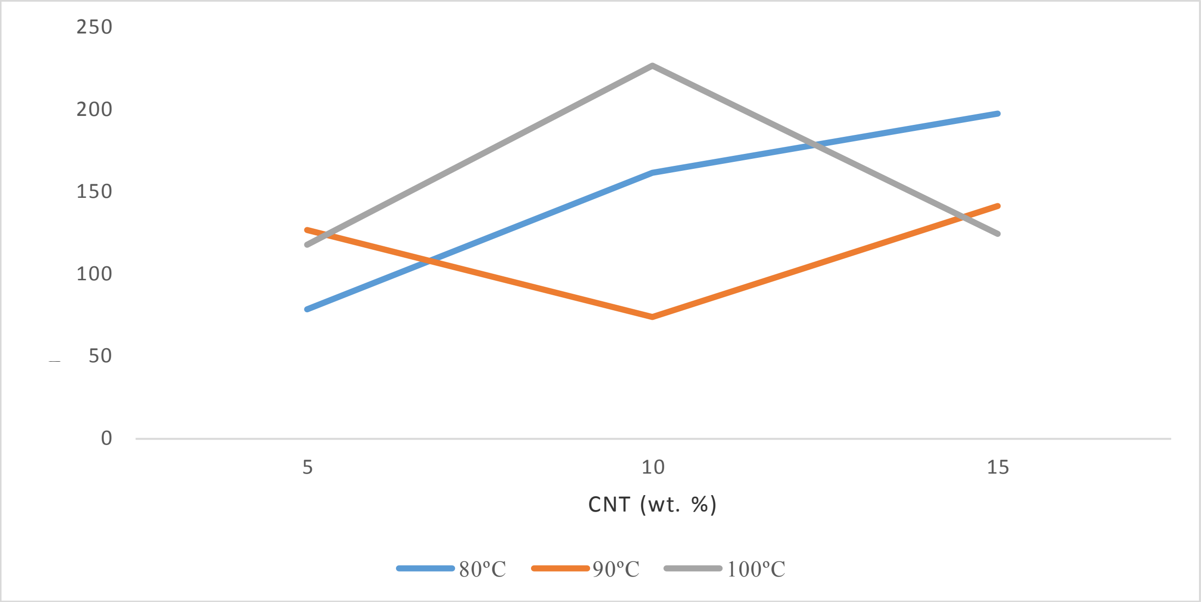


Figure 4.4 :Graph of Electrical Conductivity against content of CNT

Based on the result of electrical conductivity in Table 4.3, Figure 4.4 were plotted electrical conductivity against of CNTs to show the trend of each composites. From the Figure 4.4 it clearly show that by increases weight of CNTs composition and temperature, the electrical conductivity was increased. At temperature $90\ ^{\circ}C$ the electrical conductivity dropped at 10% weight of CNTs, but it rise up at 15% weight of CNTs. Meanwhile, when the temperature $100\ ^{\circ}C$, electrical conductivity rise up at 10% weight of CNTs, but it dropped at 15% weight of CNTs.

4.3.3 FLEXURE RESULT

The Flexural strength was measured by the three point method using the Instron Universal Testing Machine. The specimen of rectangular cross section with a width 13mm, thickness of 3mm and the length of 100 mm (with span length equivalent of 25mm right and 25mm left) Figure 4.5 below show the length of span length. Flexure testing were conducted at ambient temperature of computer controlled Instron Universal Testing Machine by using 50kN load and a ramp rate 0.1mm/s in the laboratory atmosphere.

Figure 4.5 : Length spread span

Table 4.4: Flexural Strength for each Sample

°C CNT	80	90	100
5	1.87	2.12	2.17
10	2.39	2.02	2.12
15	3.04	3.15	1.95

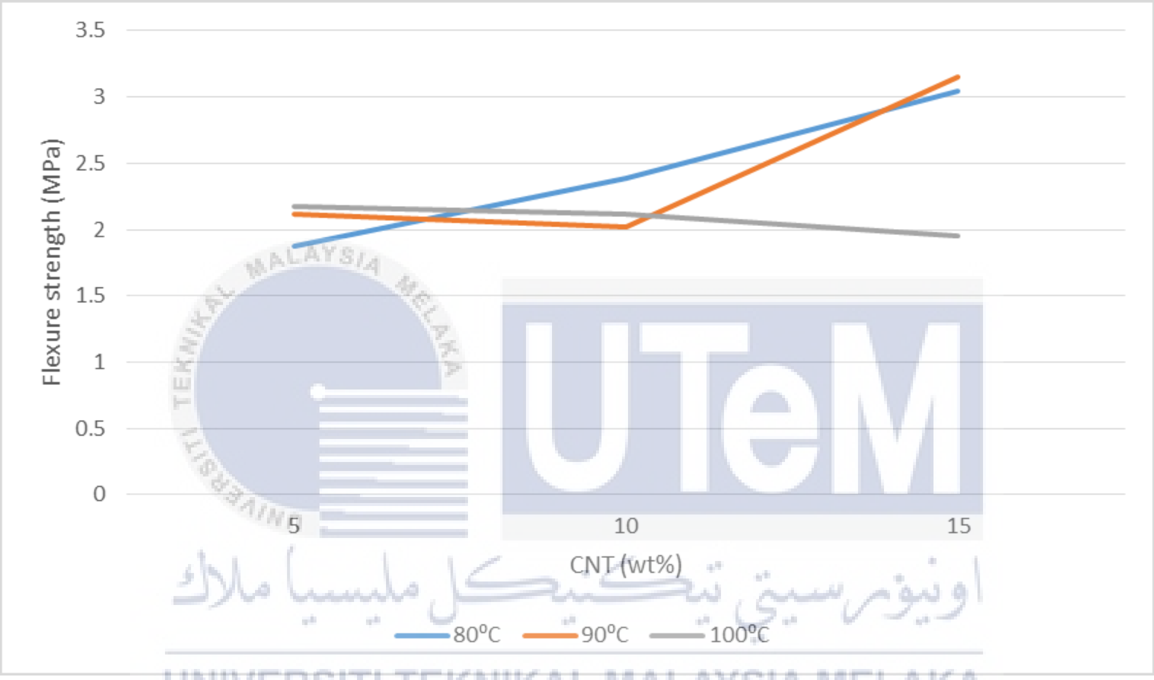


Figure 4.6: Graph of Flexural Strength against CNT wt%

The graph flexural strength against weight percentages of CNT composite with differences temperature has been plotted as shown in Figure 4.6. The flexure strength on 80°C increase gradually with increasing CNT content. Based on graph above, at temperature 90°C the flexural strength was dropped from 2.12MPa at 5wt% CNT to 2.02MPa at 10wt% CNT content. Then it was climbing to 3.15MPa at 15wt% CNT. For tempereture100°C, it was suddenly decline from 2.17MPa at 5wt% CNT to 1.95MPa at 15wt% CNT. The optimum value of flexural strength obtained was 3.15MPa at temperature 90°C with 15wt % of CNT.

4.3.4 HARDNESS RESULT

Figure 4.7 shows the variation in shore hardness result based on the difference temperature. At 80°C, the shore hardness increased from 39.73 at 5wt% to 45.55 at 10wt% then it decreases to 41.1 at 15wt% CNT. Same as temperature 80°C, at the temperature 90°C it increased from 37.78 at 5wt% to 40.2 at 10wt% CNT, then it was dropped to 34.09 at 15wt%CNT. Meanwhile, temperature 100°C it fell down from 38.32 at 5wt% to 33.22 at 15wt% CNT.

Table 4.5: Hardness Data

°C / CNT	80	90	100
5	39.73	37.78	38.32
10	45.55	40.2	35.32
15	41.1	34.09	33.22

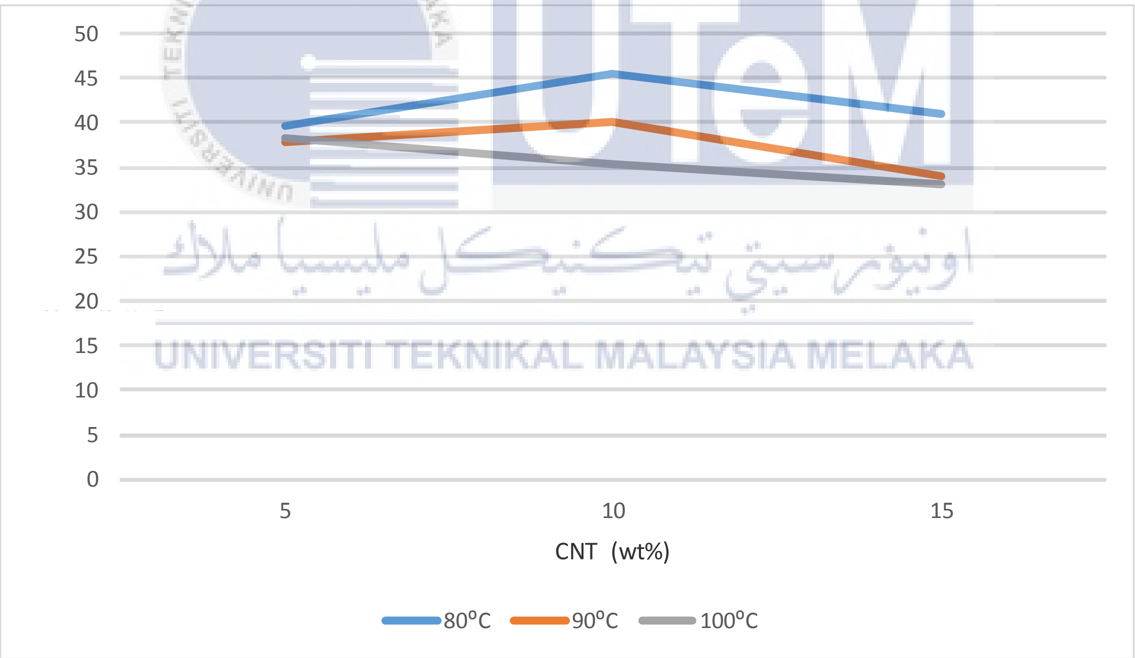


Figure 4.7: Graph of Hardness against CNT wt%

4.3.5 DENSITY RESULT

As shows in Figure 4.8, the density result shows that when temperature 80°C it declined drastically from 1.804 g/cm^3 at 5wt% to 1.637 g/cm^3 at 15wt% of CNT contained. Besides that, at temperature 90°C it also going down from 1.773 g/cm^3 at 5wt% to 1.602 g/cm^3 at 15wt% CNT. However, at 100°C at 5wt% the density suddenly grown from 1.694 g/cm^3 to 1.713 g/cm^3 at 10wt% then it slightly down to 1.569 g/cm^3 at 15wt% of CNT.

Table 4.6: Density Data for each temperature

°C **CNT**	**80**	**90**	**100**
5	1.804	1.773	1.694
10	1.731	1.723	1.713
15	1.637	1.602	1.569

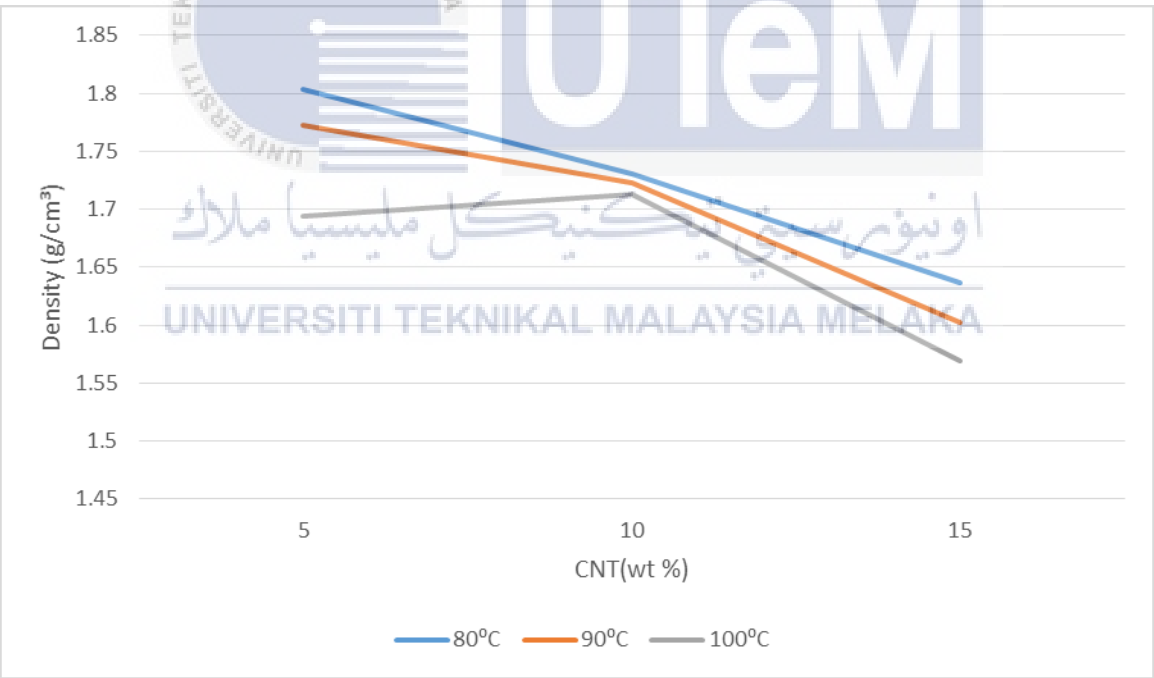


Figure 4.8: Graph of density against CNT wt%

4.3.6 MICROSTRUCTURE

This instrument forms images with electrons instead of light allowing high magnification photographs with excellent depth of field. Scanning electron microscopy (SEM) is useful for failure analysis since it can reveal where a fracture started, how fast it propagated, and whether the fracture mode was ductile or brittle. Cross sectional measurements of plating thickness (ASTM B748) can also be made with this instrument. The results are in the form of black and white images with our interpretation of their meaning**.**

Table 4.7 : 500 magnificent of SEM result

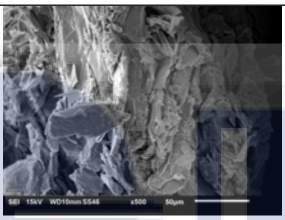
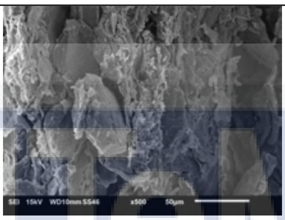
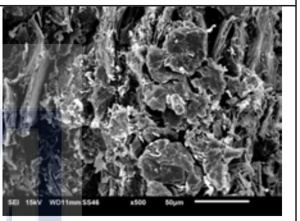
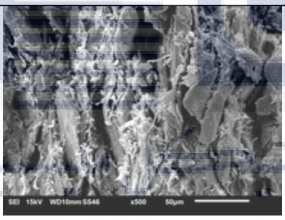
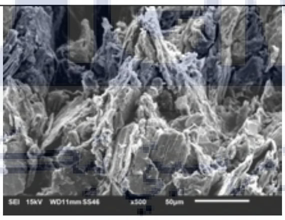
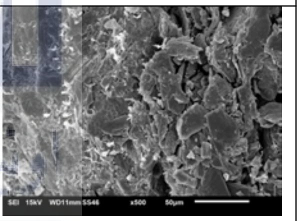
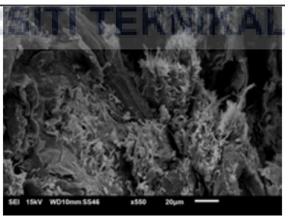
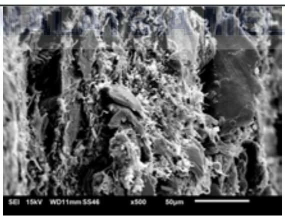
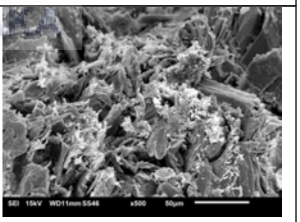
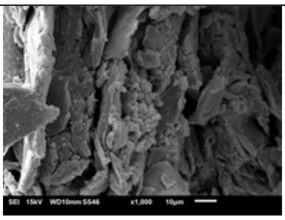
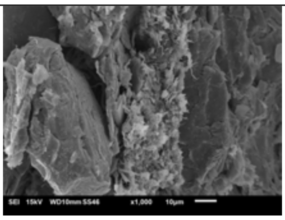
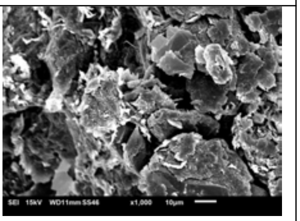
ºC **CNT**	**80**	**90**	**100**
5			
10			
15			

Table 4.8: 1000 magnificant of SEM result

ºC **CNT**	**80**	**90**	**100**
5			

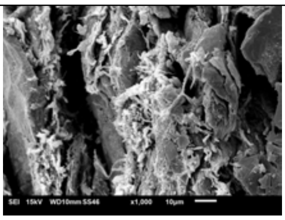
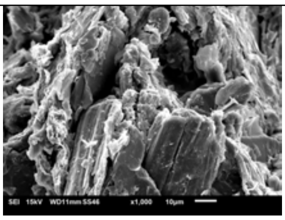
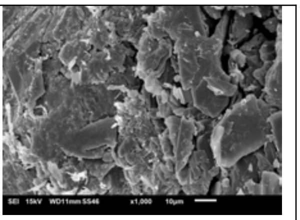
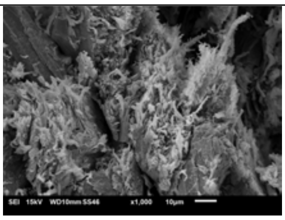
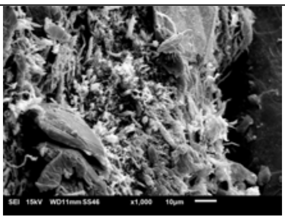
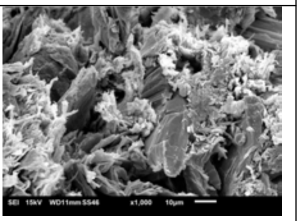
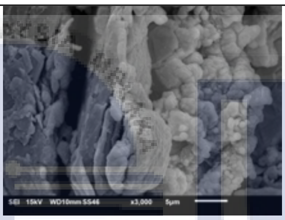
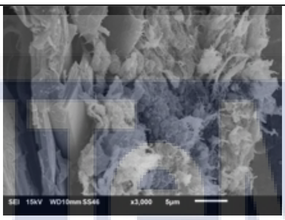
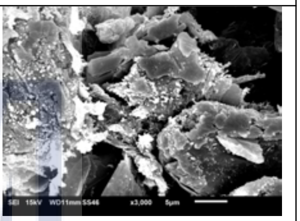
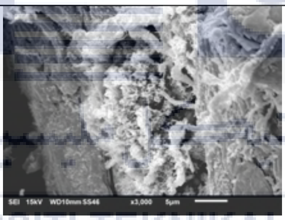
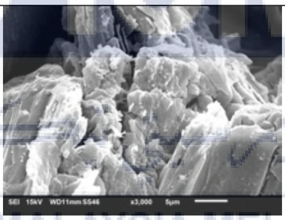
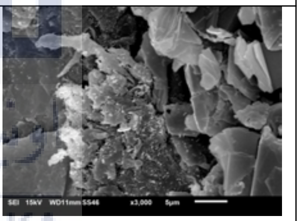
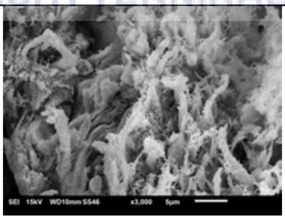
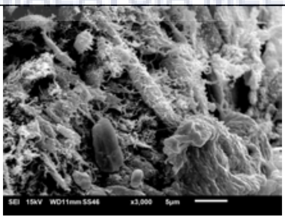
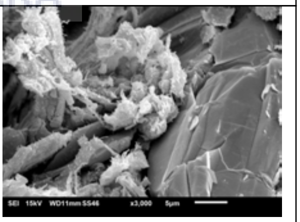
10			
15			

Table 4.9: 3000 Magnificant of SEM result

ºC **CNT**	**80**	**90**	**100**
5			
10			
15			

CHAPTER 5

DISCUSSION

5.1 PROBLEM OCCUR.

There are several problem occurs in this study. The Details of the problem will be discussed in the following sub-chapter.

5.1.1 Material stick on the mould

In the first trial of fabrication, sample difficult to be removed from the mould. It take time to remove but it will cause damage to the surface or cracks. It may be happened because of a surface that is not smooth or has not been cleaned properly. So, to overcome this problem, methods used to solve this problem is to clean the surface using isotone before and after moulding process. Other than that, by using Mylar paper (CODE). By using Mylar Film Paper it make smoother surface and easier sample to separate sample and mould.

5.2 DISCCUSION OF RESULT

There are several tests has been conducted such as electrical conductivity, flexure test, density test, hardness and microstructure analysis.The related tests were conducted to determine the effect CNT loading and temperatures (80 C, 90 C and 100 C) during the fabrication of Gr/CB/CNT/EP composite.

5.2.1 Electrical Conductivity

This is the most important properties and characteristic in bipolar plate. Bipolar plate is used as an electrical connection between two electrodes with opposite polarities. Therefore, it will implement the serial addition of the electrochemical potential of different cells in the fuel cell. In this study, the effect of CNTs loading on the electrical conductivity by adding the amount of Gr, CB, and EP. The ratio of filler and binder is fixed at75:25. The amount of CB and EP are fixed at 25% with difference temperatures (80 ° C, 90 ° C and 100 ° C) are shown in Figure 4.3. The effect of CNTs loading in electrical conductivity are, when the weight of CNTs increase, the electrical conductivity also increase. These result shown that by adding the CNTs has produces a higher of electrical conductivity but it drooped at 15 wt% of CNTs. As a general the electrical conductivity was increased with the increment of temperatures used. At the temperature 90 ° C the electrical conductivity dropped at 10% weight of CNTs, but it rise up at 15 wt% of CNTs. Meanwhile, when the temperature at 100 ° C, electrical conductivity rise up at 10% weight CNTs, but it dropped at 15 wt% of CNTs. But when it compare to the difference temperatures used, the temperature of CNTs increase, the electrical conductivity has fluctuated depending on the loading of CNTs.

5.2.2 Flexure Strength

The result of three point bending test was shown at Figure 4.4 and Table 4.5. It present the average of the nine sample. Based on the result, the effect of the loading of CNTs in flexure are, when weight of CNTs loading increase, the flexure strength also increased. Meanwhile, the temperature also give the effect of flexure strength. When temperature rise up, the flexure strength is decrease with the increasing of CNTs loading. The result of flexure strength of nine sample had not achieved the US DOE target. This is based on the mod of fracture occurs either ductile and brittle failure. In the brittle failure have two types fracture that are transgranular or intergranular fracture. Therefore, microstructure analysis was carried out to determine the type of fracture.

5.2.3 Hardness

From Figure 4.7, it shows that the trend of the weight content of CNTs in the Gr/CB/EP. When the weight content of CNTs increase, the hardness of the sample increase. But it depend on the temperature occurs, when the temperature increase, the hardness has be dropped. So based on the result of hardness test, the best of CNTs loading is at 10wt%.

5.2.4 Density

The density of the first sample with 5wt% of CNTs and at 80 ° C was the higher than other sample. By increasing weight of CNTs and the temperature its shown that no effect of the composite in the bipolar plate. The average of value density of bipolar plate is $1.694\, g/cm^3$ and it achieve the requirement that stated by DOE target.

5.2.5 Microstructure Analysis

Figure 4.9, shown the Scanning Electron Microscopic (SEM). SEM is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. Based on the SEM image of the fracture surface, its shown that it is a brittle fracture. In the brittle fracture, it shown that the crack usually occurs by increased the stress. There are two type of brittle fracture, which is transgranular and intergranular. The type of brittle fracture depend on the grain boundaries are stronger or weaker than the grains. By refer to the fracture surface of SEM image, can be conclude that the bonding of the CNTs were weak on the interface of CNTs and the matric. The crack deflection occurred along this weak interface. So, when done the fracture test, the result it shown that very low value and out of the DOE target.

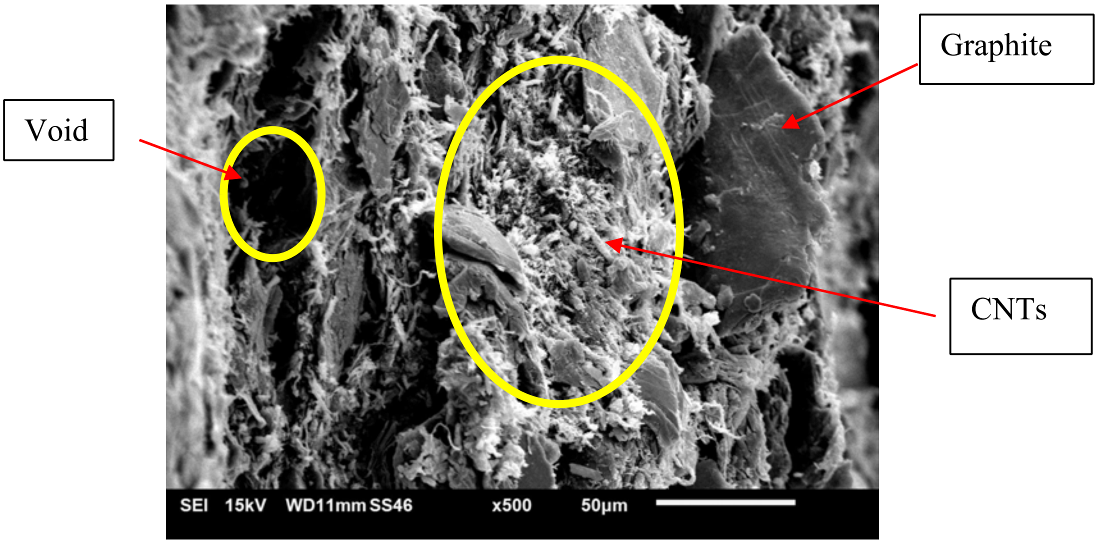


Figure 4.9: SEM image 10 wt% CNTs at $90^{\circ} c$

Based on the SEM image , the effect of CNTs loading can be defined at 5wt% and 15wt% of CNTs loading as shown at Figure 4.10 and Figure 4.11. When at 5wt% loading of CNTs the result of the electrical and mechanical properties is higher than other sample. Meanwhile, at 15wt%loading of CNTs shown that the lower result of electrical and mechanical properties. Furthermore, based on this image also it shown that the percentages of void is higher.Therefor, its given the effect on the electrical and mechanical properties.

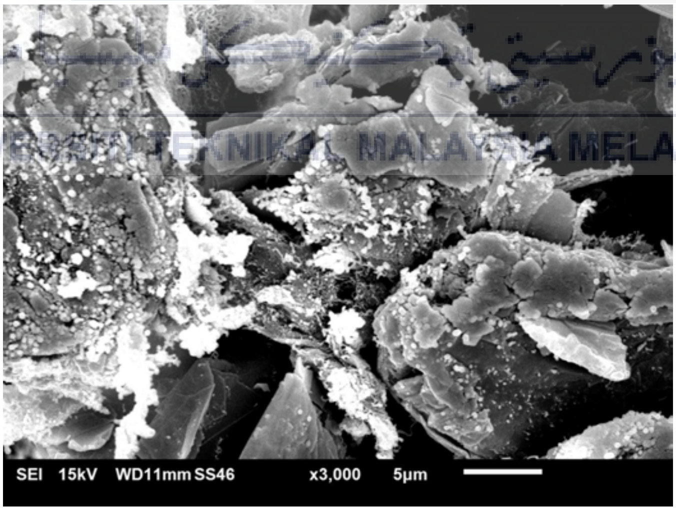


Figure 4.10 : SEM image at 5wt% loading of CNTs

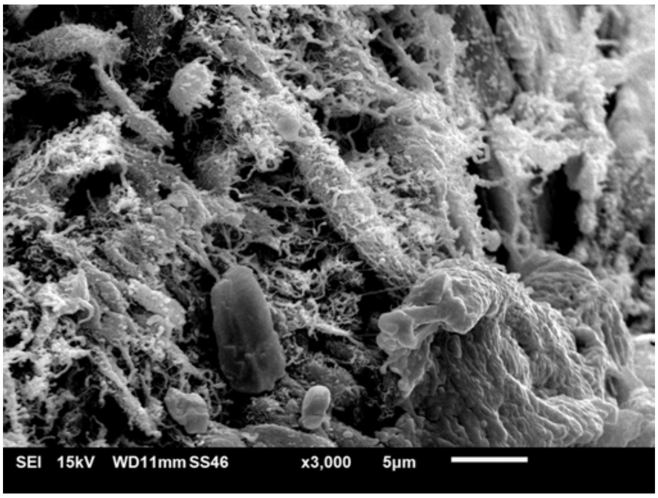


Figure 4.11: SEM image at 15wt% loading of CNTs

The temperature also a parameter that give the effect on the result. Temperature is the important parameter in compression process. The sample must fully compact well together. This phenomena give the effect on the electrical and mechanical properties. If the sample have very good compactness, the sample will reduce the percentages of void. Figure 4.12 shown that at 80 ˚ C the result of electrical conductivity and mechanical properties is low then the temperature 100˚C.

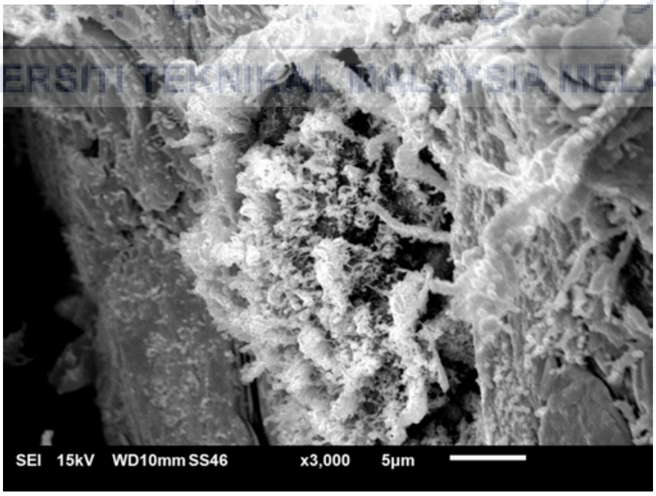


Figure 4.12 : SEM Image at 10wt% of the 80˚C

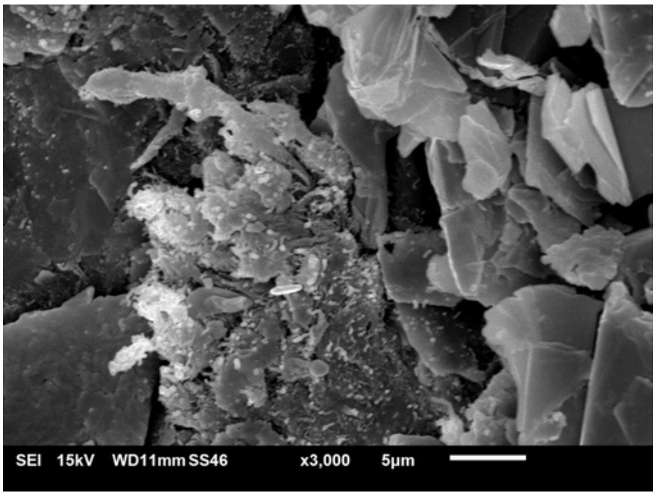


Figure 4.13 : SEM Image at 10wt% of the 100 ˚ C

CHAPTER 6

CONCLUSION AND RECOMMENDATION

6.1 CONCLUSION

There are two main objective of this research. Firstly, to study the effect of CNTs on the properties of Gr/CB/EP and to determine the critical loading of CNTs in Gr/CB/EP. Therefore, by this research was study the effect of CNT loading on the electrical and mechanical properties of Gr/CNT/CB as a filler and EP as a binder. In order to determine the effect of CNTs content in Gr/CB/CNT/EP composite, the testing such as electrical conductivity, flexure testing, density testing, hardness and microstructure analysis has been done. All nine (9) specimen are successfully fabricated and tested through this study.

Based on the experiment and the result obtained, it shown that at 10% weight of CNTs had the best achieved requirement of US DOE target on electrical conductivity, hardness and density. Only, the flexure strength not achieved the target based on the factor that need to study further and need to improved in future.

The first process fabrication of the specimen was mixing. Mixing process is the important part of the fabrication composite. It is because composite is the combination o two or more material that contain matric and reinforcement. Therefore, in the mixing process, the material must mix ideally to produce a material with characteristic or properties needed.

Other than that, the important in fabrication composite of bipolar plate are compression. Compression molding is known that the step to develop the composite specimen. Therefore, the important parameter that need to be consider in the compression process that control the fabrication process are temperature and pressure.

6.2 RECOMMENDATION

In this study, bipolar plate that containing the CNTs is under development process. Therefore, have several technique or process that need to be improve to achieve the mechanical and electrical properties. There are possible recommendation for the future that need to improve.

6.2.1 Method for mixing process

The process of mixing the Gr/CB/ CNTs with the EP is by using the manual lab blender (Panasonic). This equipment is not the best equipment in the mixing process to fabricate the specimen. By using this blender, it maybe not mix well for binder and filler. Furthermore, the temperature to be mix cannot be control and the time taken to be mix in stage of try and error. So, to solve this problem, a recommended to use the suitable mixing machine that can control temperature and time.

6.2.2 Change the ratio of filler and binder, temperature, and pressure

Based on the result of fracture obtained, maybe at the compression process the temperature and pressure is not the best. Because when the difference of temperature change the result is fractuated by increasing the weight content of CNTs. Other than that, by consider the CNTs content. Maybe should use 0-10%wt CNTs.

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APPENDIX A

ELECTRICAL CONDUCTIVITY

SAMPLE 1

A) FRONT

GR/CB/CNT/EP 45/25/5/25	**T= 80ºC**	**P=60 tan**
I(mA)	**V(mV)**	**σ**
1	0.046	54.63
1	0.035	71.81
1	0.045	55.85
1	0.039	64.44
1	0.031	81.07
1	0.038	66.14
1	0.032	78.54
1	0.031	81.07
1	0.035	71.81
		69.48302

B) BACK

GR/CB/CNT/EP 45/25/5/25	**T= 80ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.022	114.24
1	0.032	78.54
1	0.036	69.81
1	0.06	41.89
1	0.028	89.76
1	0.03	83.77
1	0.041	61.30
1	0.018	139.62
1	0.026	96.66
		86.17522

SAMPLE 2

A) FRONT

GR/CB/CNT/EP 40/25/10/25	**T= 80ºC**	**P=60 tan**
I(mA)	V(mV)	**σ**
1	0.013	193.32
1	0.019	132.27
1	0.018	139.62
1	0.014	179.51
1	0.019	132.27
1	0.018	139.62
1	0.017	147.83
1	0.019	132.27
1	0.014	179.51
		152.9157

B) BACK

GR/CB/CNT/EP 40/25/10/25	**T= 80ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.01	251.32
1	0.015	167.55
1	0.017	147.83
1	0.014	179.51
1	0.027	93.08
1	0.018	139.62
1	0.016	157.07
1	0.012	209.43
1	0.014	179.51
		169.4367

SAMPLE 3

A) FRONT

GR/CB/CNT/EP 35/25/15/25	**T= 80ºC**	**P=60 tan**
I(mA)	V(mV)	**σ**
1	0.013	193.32
1	0.011	228.47
1	0.009	279.24
1	0.017	147.83
1	0.011	228.47
1	0.013	193.32
1	0.016	157.07
1	0.013	193.32
1	0.016	157.07
		197.57

B) BACK

GR/CB/CNT/EP 35/25/15/25	**T= 80ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.013	193.32
1	0.013	193.32
1	0.011	228.47
1	0.015	167.55
1	0.013	193.32
1	0.014	179.51
1	0.015	167.55
1	0.013	193.32
1	0.01	251.32
		196.4087

SAMPLE 4

A) FRONT

GR/CB/CNT/EP 45/25/5/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	**σ**
1	0.017	147.83
1	0.021	119.68
1	0.018	139.62
1	0.013	193.32
1	0.017	147.83
1	0.011	228.47
1	0.021	119.68
1	0.029	86.66
1	0.023	109.27
		143.5958

B) BACK

GR/CB/CNT/EP 45/25/5/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.018	139.62
1	0.014	179.51
1	0.023	109.27
1	0.022	114.24
1	0.17	14.78
1	0.023	109.27
1	0.018	139.62
1	0.028	89.76
1	0.03	83.77
		108.8711

SAMPLE 5

A) FRONT

GR/CB/CNT/EP 40/25/10/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	**σ**
1	0.031	81.07
1	0.047	53.47
1	0.022	114.24
1	0.042	59.84
1	0.04	62.83
1	0.035	71.81
1	0.048	52.36
1	0.044	57.12
1	0.069	36.42
		65.46092

B) BACK

GR/CB/CNT/EP 40/25/10/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.033	76.16
1	0.025	100.53
1	0.042	59.84
1	0.039	64.44
1	0.031	81.07
1	0.022	114.24
1	0.021	119.68
1	0.03	83.77
1	0.056	44.88
		82.73266

SAMPLE 6

A) FRONT

GR/CB/CNT/EP 35/25/15/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.018	139.62
1	0.012	209.43
1	0.01	251.32
1	0.019	132.27
1	0.02	125.66
1	0.027	93.08
1	0.014	179.51
1	0.017	147.83
1	0.018	139.62
		157.5945

B) BACK

GR/CB/CNT/EP 35/25/15/25	**T= 90ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.014	179.51
1	0.012	209.43
1	0.017	147.83
1	0.026	96.66
1	0.035	71.81
1	0.021	119.68
1	0.028	89.76
1	0.032	78.54
1	0.019	132.27
		125.0539

SAMPLE 7

A) FRONT

GR/CB/CNT/EP 45/25/5/25	T= 100ºC	P=60 tan
I(mA)	V(mV)	σ
1	0.02	125.66
1	0.019	132.27
1	0.013	193.32
1	0.019	132.27
1	0.022	114.24
1	0.019	132.27
1	0.018	139.62
1	0.036	69.81
1	0.017	147.83
		131.9221

B) BACK

GR/CB/CNT/EP 45/25/5/25	T= 100ºC	P=60 tan
I(mA)	V(mV)	σ
1	0.019	132.27
1	0.044	57.12
1	0.017	147.83
1	0.031	81.07
1	0.029	86.66
1	0.037	67.92
1	0.028	89.76
1	0.017	147.83
1	0.021	119.68
		103.3495

SAMPLE 8

A) FRONT

GR/CB/CNT/EP 40/25/10/25	T= 100ºC	P=60 tan
I(mA)	V(mV)	σ
1	0.024	104.72
1	0.01	251.32
1	0.008	314.15
1	0.016	157.07
1	0.009	279.24
1	0.022	114.24
1	0.008	314.15
1	0.01	251.32
1	0.006	418.86
		245.0068

B) BACK

GR/CB/CNT/EP 40/25/10/25	T= 100ºC	P=60 tan
I(mA)	V(mV)	σ
1	0.008	314.15
1	0.022	114.24
1	0.011	228.47
1	0.01	251.32
1	0.01	251.32
1	0.026	96.66
1	0.01	251.32
1	0.018	139.62
1	0.011	228.47
		208.3956

SAMPLE 9

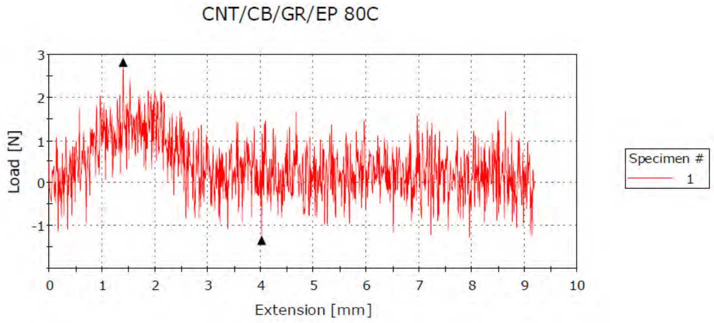
A) FRONT

GR/CB/CNT/EP 35/25/15/25	**T= 100ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.013	193.32
1	0.014	179.51
1	0.016	157.07
1	0.015	167.55
1	0.024	104.72
1	0.017	147.83
1	0.031	81.07
1	0.016	157.07
1	0.017	147.83
		148.4424

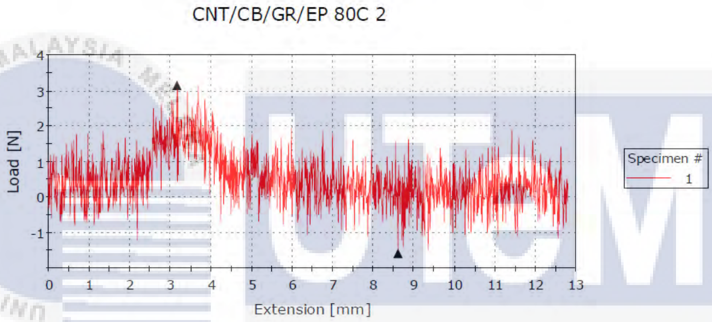
B) BACK

GR/CB/CNT/EP 35/25/15/25	**T= 100ºC**	**P=60 tan**
I(mA)	V(mV)	σ
1	0.025	100.53
1	0.031	81.07
1	0.024	104.72
1	0.021	119.68
1	0.034	73.92
1	0.029	86.66
1	0.023	109.27
1	0.027	93.08
1	0.02	125.66
		99.39724

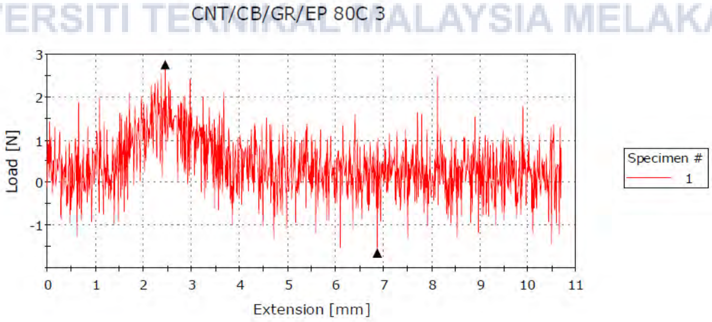
APPENDIX B
FLEXURE RESULT



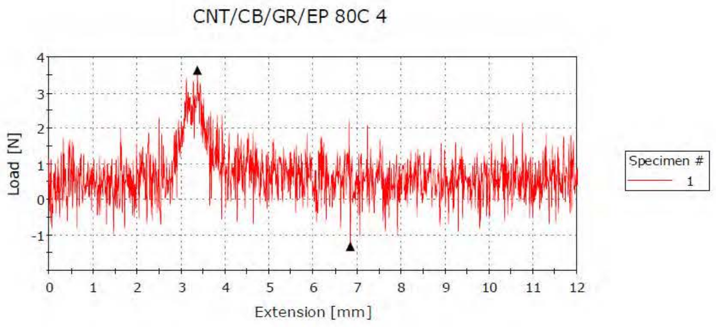
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.82	1.81	1.40	0.00



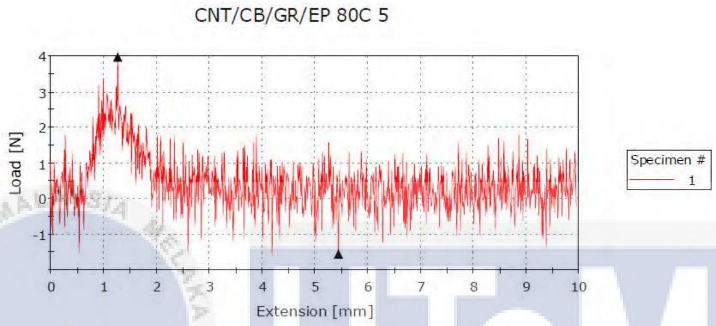
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.15	2.02	3.16	0.00



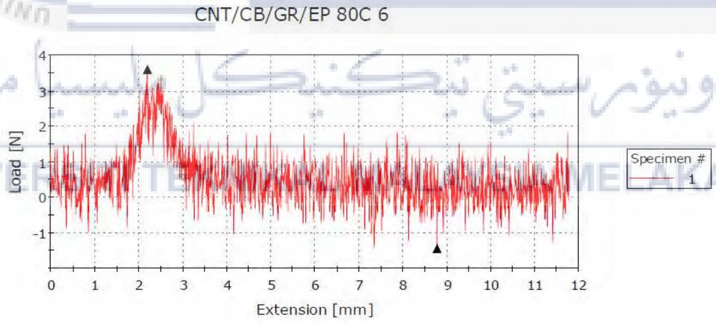
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.75	1.77	2.45	0.00


CNT/CB/GR/EP 80C 4

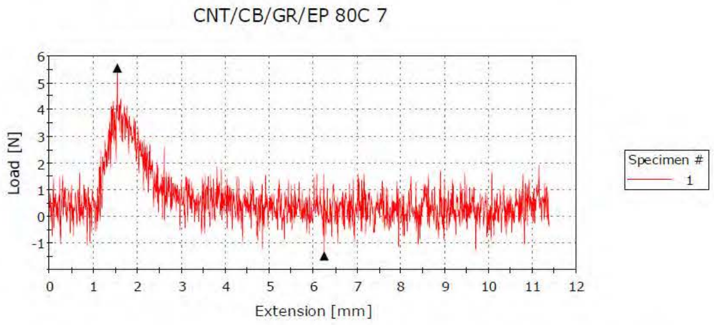
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.62	2.32	3.37	0.00


CNT/CB/GR/EP 80C 5

	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.96	2.54	1.27	0.00

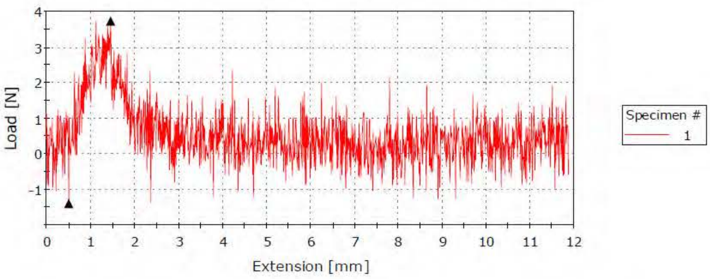

CNT/CB/GR/EP 80C 6

	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.60	2.30	2.20	0.00


CNT/CB/GR/EP 80C 7

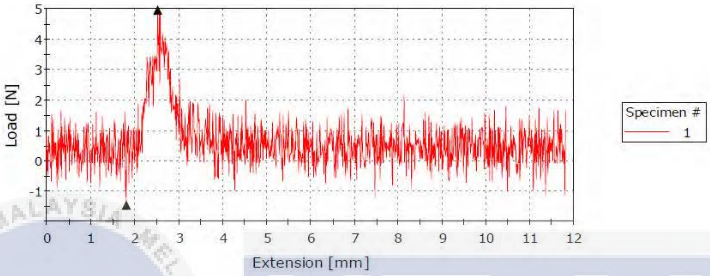
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	5.55	3.56	1.55	0.00

CNT/CB/GR/EP 80C 8



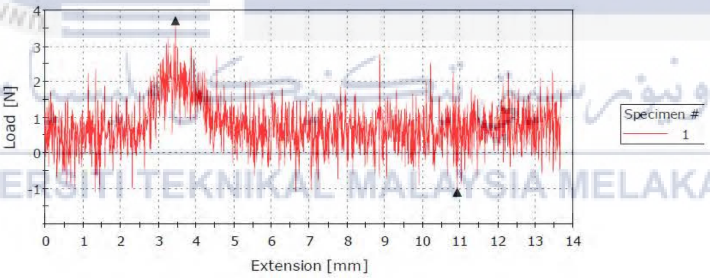
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.73	2.39	1.46	0.00

CNT/CB/GR/EP 80C 9



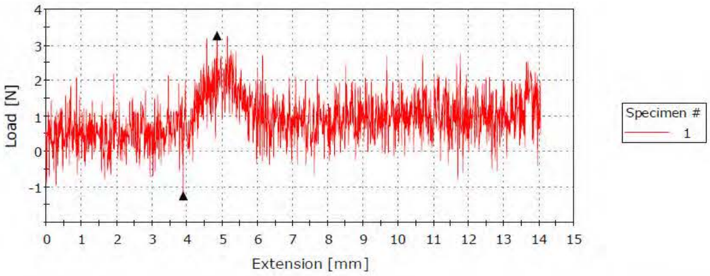
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	4.95	3.17	2.52	0.00

CNT/CB/GR/EP 80C 10



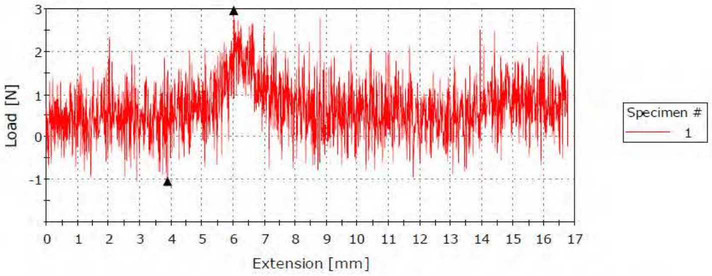
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.71	2.38	3.45	0.00

CNT/CB/GR/EP 80C 11



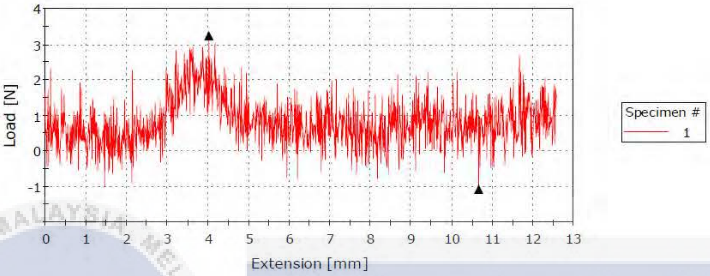
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.25	2.09	4.85	0.00

CNT/CB/GR/EP 80C 12



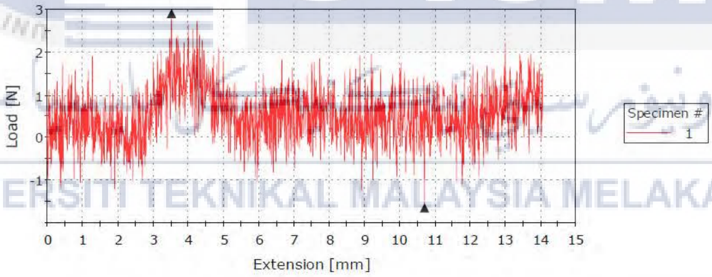
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.97	1.90	6.02	0.00

CNT/CB/GR/EP 80C 13



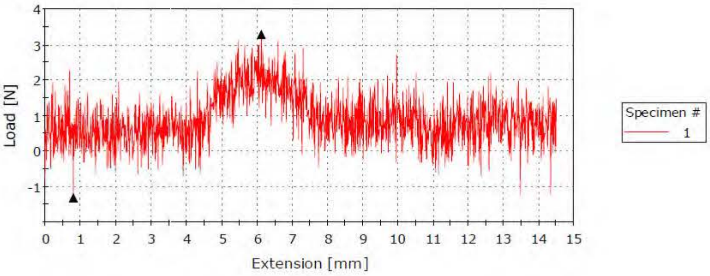
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.24	2.08	4.02	0.00

CNT/CB/GR/EP 80C 14



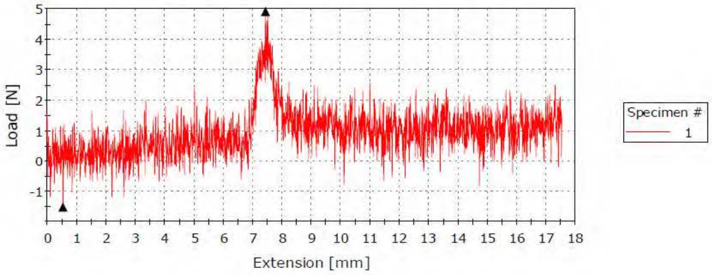
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.91	1.86	3.52	0.00

CNT/CB/GR/EP 80C 15



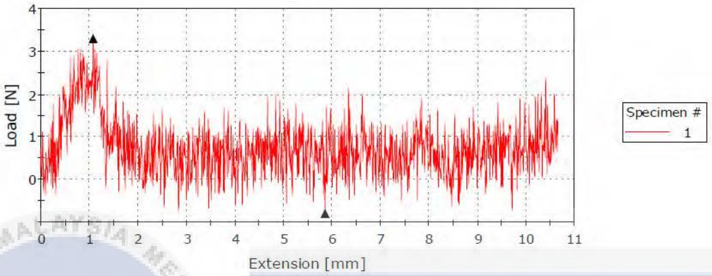
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.29	2.11	6.11	0.01

CNT/CB/GR/EP 80C 16



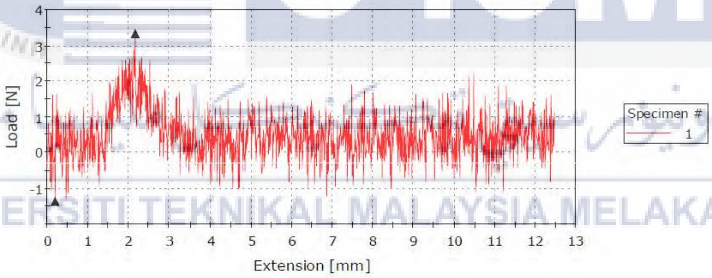
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	4.91	3.15	7.43	0.00

CNT/CB/GR/EP 80C 19



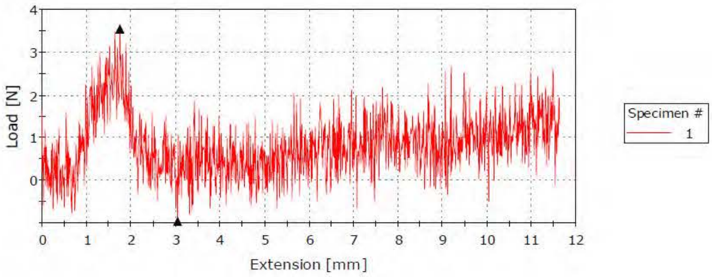
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.30	2.12	1.08	0.00

CNT/CB/GR/EP 80C 20



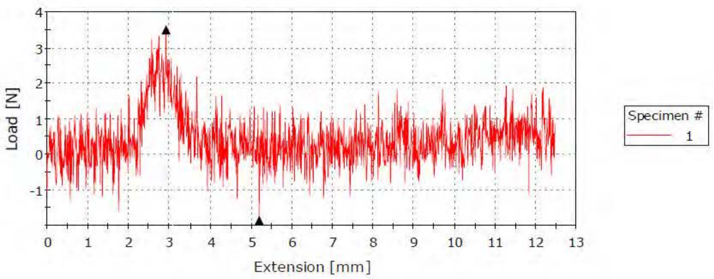
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.33	2.14	2.17	0.00

CNT/CB/GR/EP 80C 21



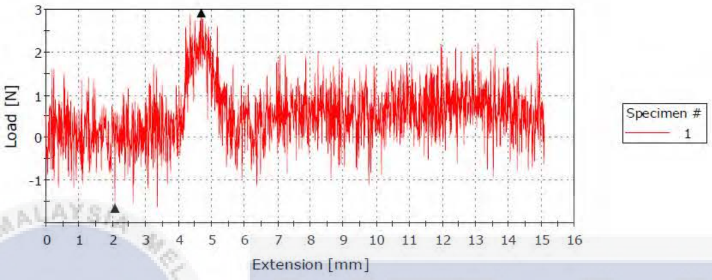
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.55	2.27	1.75	0.00

CNT/CB/GR/EP 80C 21



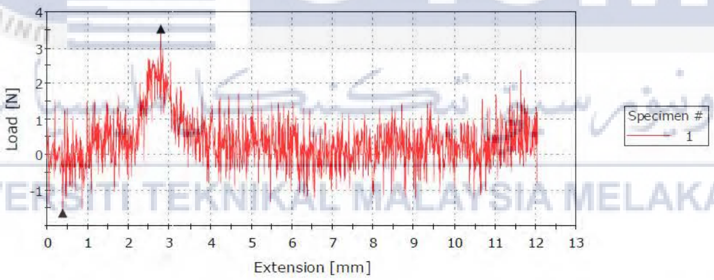
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.49	2.24	2.92	0.00

CNT/CB/GR/EP 80C 23



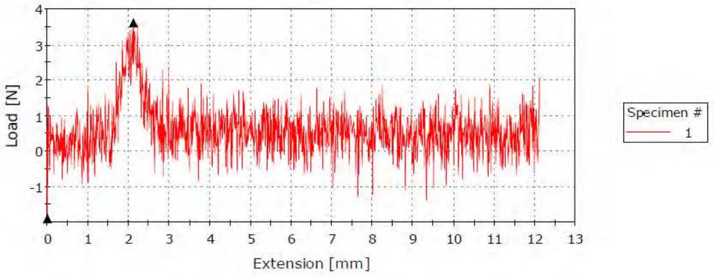
	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.92	1.87	4.69	0.00

CNT/CB/GR/EP 80C 24

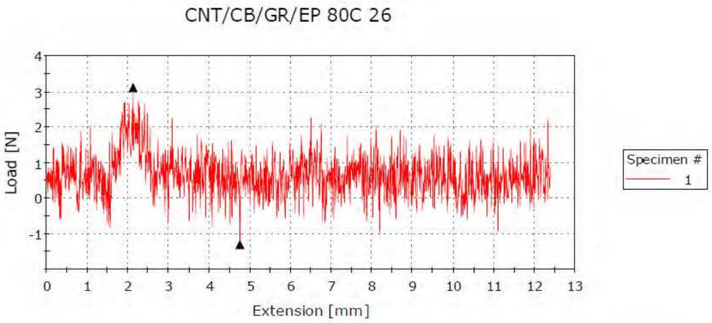


	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.52	2.25	2.79	0.00

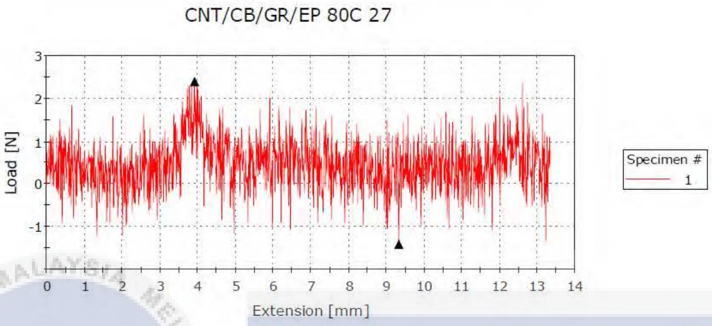
CNT/CB/GR/EP 80C 25



	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.61	2.31	2.12	0.00



	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	3.11	1.99	2.13	0.00



	Maximum Load [N]	Flexure stress at Maximum Load [MPa]	Extension at Maximum Load [mm]	Energy at Maximum Load [J]
1	2.40	1.54	3.92	0.00

Designation: C 611 – 98 (Reapproved 2005)$^{\epsilon 1}$

An American National Standard

Standard Test Method for
Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature[1]

This standard is issued under the fixed designation C 611; 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.

ϵ^1 NOTE—Editorial changes were made to Figure 3 in May 2005.

1. Scope

1.1 This test method covers the determination of the electrical resistivity of manufactured carbon and graphite articles at room temperature.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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. Terminology

2.1 *Definition:*

2.1.1 *resistivity*—the property of a material that determines its resistance to the flow of an electrical current. It is defined as the value of ρ, in milliohm metres, as follows:

$$\rho = (R \cdot A)/L$$

where:

R = resistance of a specimen of the material of uniform cross section, ohms,
A = uniform cross section, mm^2, and
L = distance between potential contacts, mm.

2.1.1.1 In cases where resistivity is requested in ohm-inches, multiply ρ in milliohm metres by 0.03937.

3. Significance and Use

3.1 This test method provides a means of determining the electrical resistivity of carbon or graphite specimens. The use of specimens that do not conform to the specimen size limitations described in the test method may result in an alteration of test method accuracy.

4. Apparatus

4.1 The means for applying current and potential terminals to the specimen is specified in 5.2.3.1. A typical specimen holder is shown in Fig. 1.

4.2 *Bridge*, *Potentiometer*, or *Suitable Digital Voltmeter*, with necessary accessories for making resistance measurements with a limit of error of less than 0.5 %. Fig. 2 schematically depicts two wiring diagrams that have been found satisfactory for this purpose.

4.3 The means for measuring the dimensions of the specimen should be adequate to determine its gage length and its mean area of cross section, each within 0.5 %.

5. Test Specimen

5.1 The test specimen may be in the form of a strip, rod, bar, or tube.

5.2 In order to determine the resistivity, each specimen shall conform to the following:

5.2.1 The cross-sectional area shall be uniform within 0.75 %. In general, the diameter of circular cross section, or the thickness and width of a strip specimen shall be determined by micrometer measurements, and a sufficient number of measurements shall be made to obtain a mean cross-sectional area to within 0.5 %. The test specimen shall be machined to yield planar and parallel end faces. These faces shall be perpendicular to the specimen length to within 0.001 mm/mm. All surfaces shall have a surface finish visually comparable to 0.8 µm (32 µin.) rms. Reasonable care should be exercised to assure that all edges are sharp and without chips or other flaws.

5.2.2 The test specimen shall show no defects observable with normal vision and shall be free of surface deposits.

5.2.3 The minimum ratio of specimen length to maximum cross-sectional dimension (width or diameter) shall be 6 : 1.

5.2.3.1 The gage length may be measured by any scale that will give an accuracy of ±0.5 % in the length measured. In the direction of the length of the specimen, the dimension of each potential contact shall be not more than 0.5 % of the distance between the potential contacts. The minimum distance between each potential contact and the adjacent current contact shall be

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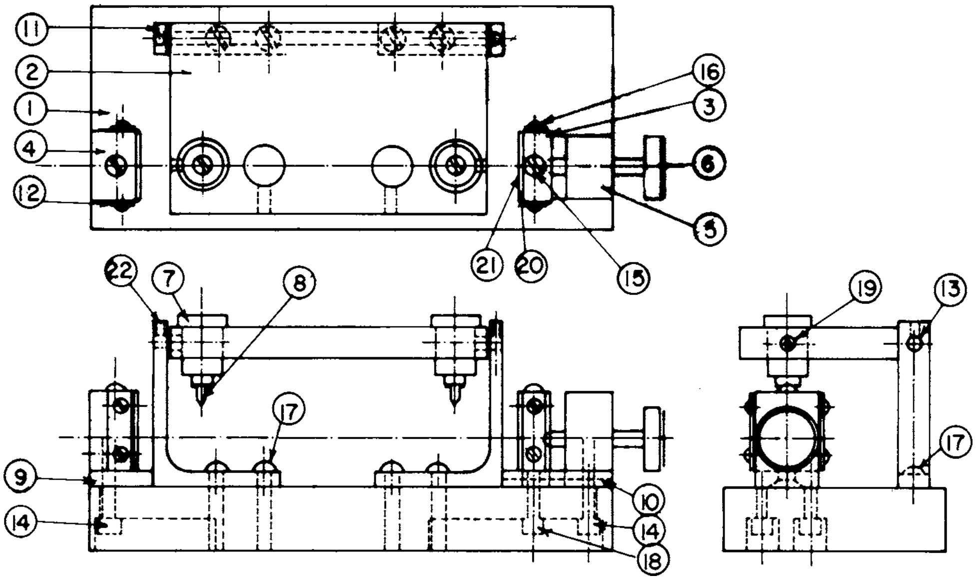
[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products .

Current edition approved May 1, 2005. Published May 2005. Originally approved in 1969. Last previous edition approved in 1998 as C 611 – 98.



1—Base block
2—Pivot block
3—Current block adjustable
4—Current block stationary
5—Clamp block
6—Clamp screw
7—Brush holder
8—Contacts
9—Current block support
10—Current block guide
11—Pivot bracket
12—Wire gauze holder
13—Pivot red
14—Screw: sockethead
15—Roundhead screw
16—Roundhead screw
17—Roundhead screw
18—Screw, sockethead
19—Set screw
20—Neoprene
21—Wire gauze
22—Set screw

NOTE—Contacts for the voltage and current probes may be made through channels drilled in the brush holders (*7*) and the current blocks (*3* and *4*), respectively.

FIG. 1 Typical Test Apparatus

the maximum cross-sectional dimension (width or diameter) of the specimen. If knife edges are used, they shall be parallel to each other and perpendicular to the longitudinal direction of the sample. The minimum ratio of gage length to maximum cross-sectional dimension (width or diameter) shall be 4 : 1.

5.2.4 No dimension shall be smaller than five times the length of the largest visible particle.

5.2.5 No joints or splices are permissible, unless this is the variable under study.

6. Conditioning

6.1 The specimen shall be dried for a minimum of 2 h at 110°C, cooled to room temperature in a desiccator, and stored in a desiccator until tested.

7. Procedure

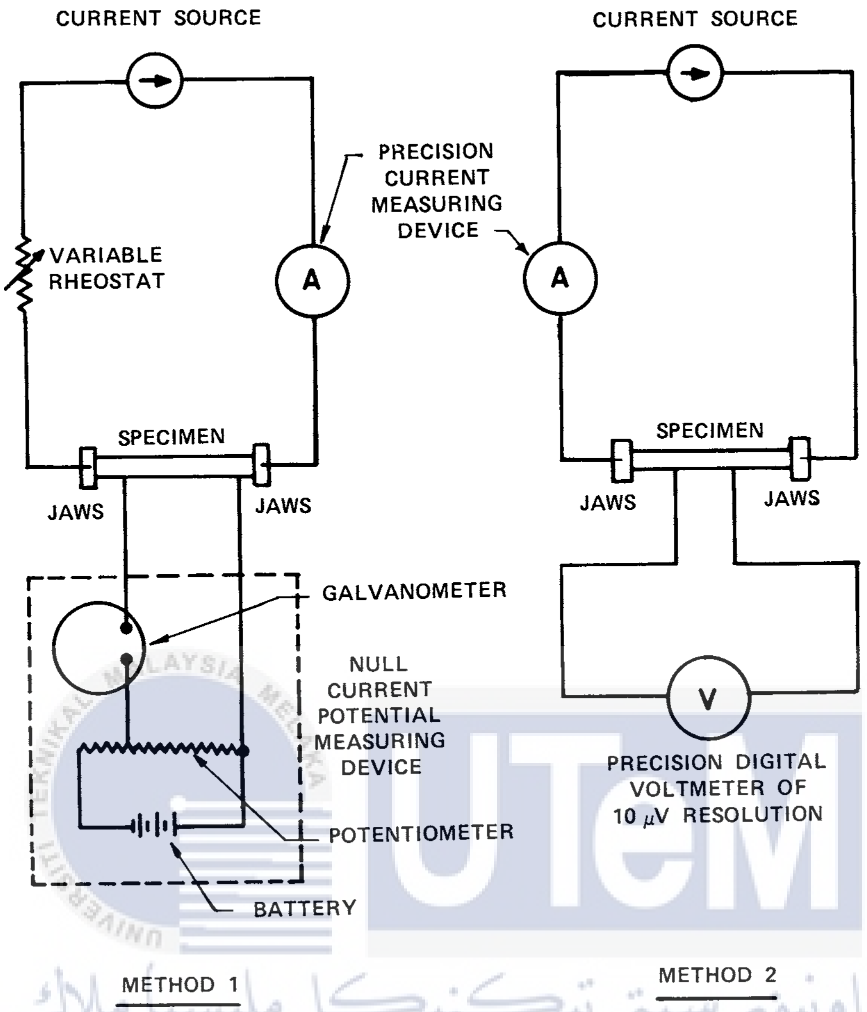
7.1 *Resistance Measurement*—Measure resistance with instruments accurate to ±0.5 % or less (see Note 1). To ensure a correct reading, the reference standard and the test specimen must be allowed to come to the same temperature as the surrounding medium.

NOTE 1—For resistance below 10 Ω, a Kelvin bridge method may be used, and for higher resistance, a Wheatstone bridge method may be used.

7.1.1 Clean the surface of the specimen at current and potential contact points to obtain good electrical contact. Mount the sample in the test apparatus, apply current, and measure the voltage. Take four measurements, on each side of a rectangular specimen, or at 90° (π/2 radians) apart on a round specimen. Reverse the current direction and take four measurements again. Remove the specimen from the test apparatus, turn it end for end, replace it in the apparatus, and repeat the measurements. The total of 16 measurements is recommended to minimize errors due to contact potential and forward and reverse currents. Average all individual values of measured resistance and use this value to calculate the resistivity.

7.2 *Heating of Specimen*—In all resistance measurements, the measuring current raises the temperature of the specimen above that of the surrounding medium. Therefore, take care to keep the magnitude of the current low, and the time short enough, so that changes in resistance cannot be detected. The measuring current shall be so small that the resistance of a specimen is not changed, thereby, as much as 0.1 %. This condition may be determined experimentally, or calculated from the power expended and the surface area of the specimen. A specimen heating check should be run after each group of samples. If resistance change exceeds 0.1 %, the sample should be cooled to ambient temperature and rerun at a lower measuring current.

7.3 A sample data collection work sheet that may be used for the testing is shown in Fig. 3.



NOTE—Adjustable, regulated dc power supply—line and load regulation to 0.1 % and ripple and noise ≤ 0.1 %.

FIG. 2 Typical Schematic for Resistivity Measurements

8. Report

8.1 Report the following:

8.1.1 Identification and previous history of the test specimen,

8.1.2 Sample orientation,

8.1.3 Temperature of surrounding medium,

8.1.4 Dimensions of specimen used,

8.1.5 Method of measuring resistance, including gage length and probe location,

8.1.6 Value of resistance or potential plus the current readings, and

8.1.7 Calculated value of resistivity.

9. Precision and Bias [2]

9.1 A round-robin test series was run to determine the precision and bias. The results of evaluating 20 test specimens of two different grades from 9 laboratories are as follows:

Within-Lab variability	0.75 %
Between-Lab variability	2.5 %

9.2 The within-lab variability is a combination of both test error and material variability since repetitious measurements were not made on single specimens within a laboratory. Material variability was, however, minimized by normalizing the results to values averaged from consistent results from five Laboratories. This yielded the estimate of a fairly small within-laboratory variability from 0.5 to 0.75 % which still includes a minor material variability.

9.3 Homogeneity of variance by the sensitive Barlett's test was not indicated, most likely, because of the very small within-laboratory variance and sensitivity to non-normality.

9.4 The between-Lab variability estimation was made on the measurement of the same specimen between Laboratories with the obvious exception of the results from laboratory A. The results still included some material variability as the resistivity varies to some extent along the length of the specimens.

9.5 The between-lab variability is fairly small and is probably a result of a minor lack of precision in the length

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[2] Supporting data giving complete results of the round-robin testing have been filed with ASTM International Headquarters.

Date ____________
Operator ____________

Sample ID ________________________________ Sample History ________________________________

L, gage length = ____________ mm I, applied current = ____________ amps T, ambient temperature = ____________ °C

1. *Cross-Sectional Area Determination, A:*

	Diameter, mm	or	Width, mm	Thickness, mm
1.	________		________	________
2.	________		________	________
3.	________		________	________
4.	________		________	________
5.	________		________	________
$\bar{X}$ =	________		________	________

Area for rounds:

$A = \bar{X}^2_{\text{diameter}}(\pi) \div 4 =$ ________ mm^2

Area for rectangles:

$A = \bar{X}_{\text{width}}(\bar{X}_{\text{thickness}}) =$ ________ mm^2

2. *Resistance Measurement, R:*

A. Ohmmeter Technique Probe Position ________________________________

	Sample Position	*Forward Current*	*Reverse Current*
Sample Orientation 1	1	________ mΩ	________ mΩ
	2	________ mΩ	________ mΩ
	3	________ mΩ	________ mΩ
	4	________ mΩ	________ mΩ
Sample Orientation 2 (Note 3)	1	________ mΩ	________ mΩ
	2	________ mΩ	________ mΩ
	3	________ mΩ	________ mΩ
	4	________ mΩ	________ mΩ

$R_{\bar{x}}$ of all resistance measurements = ________ mΩ

B. Voltmeter Technique Probe Position ________________________________

	Sample Position	*Forward Current*	*Reverse Current*
Sample Orientation 1	1	________ mv	________ mv
	2	________ mv	________ mv
	3	________ mv	________ mv
	4	________ mv	________ mv
Sample Orientation 2 (Note 3)	1	________ mv	________ mv
	2	________ mv	________ mv
	3	________ mv	________ mv
	4	________ mv	________ mv

$V_{\bar{x}}$ of all voltage measurements = ________ mv

$R_{\bar{x}} = V_{\bar{x}} \div I =$ ________ Ω

C. Specimen Heating Check

$R_1 A - R_1 = \Delta R_1$ ________

3. *Resistivity, ρ, Calculation:*

$\rho = R_{\bar{x}}(A) \div L =$ ________ μΩm

NOTE 1—The sample history, ambient temperature, and probe position should be recorded for each sample on a separate sheet.

NOTE 2—A specimen heating check should be run after each group of samples. If resistance change exceeds 0.1 %, the sample should be cooled to ambient temperature and rerun at a lower measuring current.

NOTE 3—Remove the specimen from test apparatus, turn end for end and replace the specimen in the test apparatus.

FIG. 3 Electrical Resistivity Worksheet

measurement between voltage contacts on the specimen. This small variability could be further reduced by the use of a uniform standard specimen used to periodically check the resistivity measurement apparatus. The results were essentially unchanged over the range of 17 to 41 micro-ohm metres (700 to 1700 micro-ohm inches) in electrical resistivity.

9.6 In effect, the overall conclusion is that this test method will yield repeatable test results giving a good estimation of the electrical resistivity of a material as intended by the standard method of test.

Designation: D 790 – 03

Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[1]

This standard is issued under the fixed designation D 790; 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 These test methods cover the determination of flexural properties of unreinforced and reinforced plastics, including high-modulus composites and electrical insulating materials in the form of rectangular bars molded directly or cut from sheets, plates, or molded shapes. These test methods are generally applicable to both rigid and semirigid materials. However, flexural strength cannot be determined for those materials that do not break or that do not fail in the outer surface of the test specimen within the 5.0 % strain limit of these test methods. These test methods utilize a three-point loading system applied to a simply supported beam. A four-point loading system method can be found in Test Method D 6272.

1.1.1 *Procedure A*, designed principally for materials that break at comparatively small deflections.

1.1.2 *Procedure B*, designed particularly for those materials that undergo large deflections during testing.

1.1.3 Procedure A shall be used for measurement of flexural properties, particularly flexural modulus, unless the material specification states otherwise. Procedure B may be used for measurement of flexural strength only. Tangent modulus data obtained by Procedure A tends to exhibit lower standard deviations than comparable data obtained by means of Procedure B.

1.2 Comparative tests may be run in accordance with either procedure, provided that the procedure is found satisfactory for the material being tested.

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

1.4 *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.*

NOTE 1—These test methods are not technically equivalent to ISO 178.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]
D 638 Test Method for Tensile Properties of Plastics[2]
D 883 Terminology Relating to Plastics[2]
D 4000 Classification System for Specifying Plastic Materials[3]
D 5947 Test Methods for Physical Dimensions of Solid Plastic Specimens[4]
D 6272 Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending[4]
E 4 Practices for Force Verification of Testing Machines[5]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

3. Terminology

3.1 *Definitions*—Definitions of terms applying to these test methods appear in Terminology D 883 and Annex A1 of Test Method D 638.

4. Summary of Test Method

4.1 A bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midway between the supports (see Fig. 1). A support span-to-depth ratio of 16:1 shall be used unless there is reason to suspect that a larger span-to-depth ratio may be required, as may be the case for certain laminated materials (see Section 7 and Note 8 for guidance).

4.2 The specimen is deflected until rupture occurs in the outer surface of the test specimen or until a maximum strain (see 12.7) of 5.0 % is reached, whichever occurs first.

4.3 Procedure A employs a strain rate of 0.01 mm/mm/min [0.01 in./in./min] and is the preferred procedure for this test method, while Procedure B employs a strain rate of 0.10 mm/mm/min [0.10 in./in./min].

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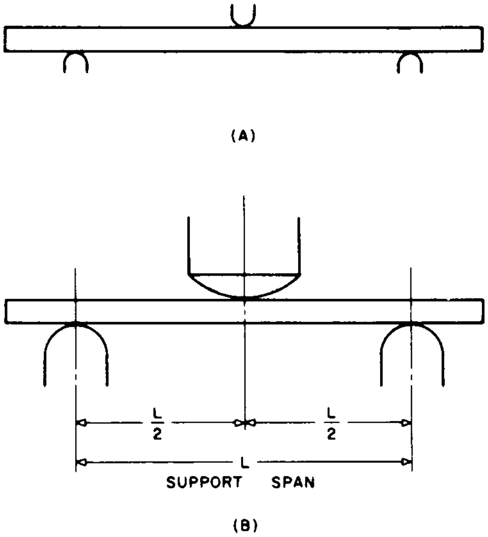
[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

Current edition approved March 10, 2003. Published April 2003. Originally approved in 1970. Last previous edition approved in 2002 as D 790 – 02.

[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 08.02.
[4] *Annual Book of ASTM Standards*, Vol 08.03.
[5] *Annual Book of ASTM Standards*, Vol 03.01.
[6] *Annual Book of ASTM Standards*, Vol 14.02.

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



NOTE—(*a*) Minimum radius = 3.2 mm [⅛ in.]. (*b*) Maximum radius supports 1.6 times specimen depth; maximum radius loading nose = 4 times specimen depth.

FIG. 1 Allowable Range of Loading Nose and Support Radii

5. Significance and Use

5.1 Flexural properties as determined by these test methods are especially useful for quality control and specification purposes.

5.2 Materials that do not fail by the maximum strain allowed under these test methods (3-point bend) may be more suited to a 4-point bend test. The basic difference between the two test methods is in the location of the maximum bending moment and maximum axial fiber stresses. The maximum axial fiber stresses occur on a line under the loading nose in 3-point bending and over the area between the loading noses in 4-point bending.

5.3 Flexural properties may vary with specimen depth, temperature, atmospheric conditions, and the difference in rate of straining as specified in Procedures A and B (see also Note 8).

5.4 Before proceeding with these test methods, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters, or combination thereof, covered in the materials specification shall take precedence over those mentioned in these test methods. If there are no material specifications, then the default conditions apply. Table 1 in Classification System D 4000 lists the ASTM materials standards that currently exist for plastics.

6. Apparatus

6.1 *Testing Machine*— A properly calibrated testing machine that can be operated at constant rates of crosshead motion over the range indicated, and in which the error in the load measuring system shall not exceed ±1 % of the maximum load expected to be measured. It shall be equipped with a deflection measuring device. The stiffness of the testing machine shall be such that the total elastic deformation of the system does not exceed 1 % of the total deflection of the test specimen during testing, or appropriate corrections shall be made. The load indicating mechanism shall be essentially free from inertial lag at the crosshead rate used. The accuracy of the testing machine shall be verified in accordance with Practices E 4.

TABLE 1 Flexural Strength

Material	Mean, 10^3 psi	Values Expressed in Units of % of 10^3 psi			
		V_r[A]	V_R[B]	r[C]	R[D]
ABS	9.99	1.59	6.05	4.44	17.2
DAP thermoset	14.3	6.58	6.58	18.6	18.6
Cast acrylic	16.3	1.67	11.3	4.73	32.0
GR polyester	19.5	1.43	2.14	4.05	6.08
GR polycarbonate	21.0	5.16	6.05	14.6	17.1
SMC	26.0	4.76	7.19	13.5	20.4

[A] V_r = within-laboratory coefficient of variation for the indicated material. It is obtained by first pooling the within-laboratory standard deviations of the test results from all of the participating laboratories: $Sr = [[(s_1)^2 + (s_2)^2 \ldots + (s_n)^2]/n]$ $_{1/2}$ then $V_r = (S_r$ divided by the overall average for the material) × 100.

[B] V_r = between-laboratory reproducibility, expressed as the coefficient of variation: $S_R = \{S_r^2 + S_L^2\}^{1/2}$ where S_L is the standard deviation of laboratory means. Then: $V_R = (S_R$ divided by the overall average for the material) × 100.

[C] r = within-laboratory critical interval between two test results = 2.8 × V_r.

[D] R = between-laboratory critical interval between two test results = 2.8 × V_R.

6.2 *Loading Noses and Supports*—The loading nose and supports shall have cylindrical surfaces. In order to avoid excessive indentation, or failure due to stress concentration directly under the loading nose, the radii of the loading nose and supports shall be 5.0 ± 0.1 mm [0.197 ± 0.004 in.] unless otherwise specified or agreed upon between the interested clients. When other loading noses and supports are used they must comply with the following requirements: they shall have a minimum radius of 3.2 mm [⅛ in.] for all specimens, and for specimens 3.2 mm or greater in depth, the radius of the supports may be up to 1.6 times the specimen depth. They shall be this large if significant indentation or compressive failure occurs. The arc of the loading nose in contact with the specimen shall be sufficiently large to prevent contact of the specimen with the sides of the nose (see Fig. 1). The maximum radius of the loading nose shall be no more than 4 times the specimen depth.

NOTE 2—Test data have shown that the loading nose and support dimensions can influence the flexural modulus and flexural strength values. The loading nose dimension has the greater influence. Dimensions of the loading nose and supports must be specified in the material specification.

6.3 *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: a contact measuring pressure of 25 ± 2.5 kPa [3.6 ± 0.36 psi], a movable circular contact foot 6.35 ± 0.025 mm [0.250 ± 0.001 in.] in diameter and 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 [0.002 in.] over the entire foot area. Flatness of foot and anvil shall conform to the portion of the Calibration section of Test Methods D 5947.

7. Test Specimens

7.1 The specimens may be cut from sheets, plates, or molded shapes, or may be molded to the desired finished dimensions. The actual dimensions used in Section 4.2, Calculation, shall be measured in accordance with Test Methods D 5947.

NOTE 3—Any necessary polishing of specimens shall be done only in the lengthwise direction of the specimen.

7.2 *Sheet Materials (Except Laminated Thermosetting Materials and Certain Materials Used for Electrical Insulation, Including Vulcanized Fiber and Glass Bonded Mica)*:

7.2.1 *Materials 1.6 mm [1⁄16 in.] or Greater in Thickness*—For flatwise tests, the depth of the specimen shall be the thickness of the material. For edgewise tests, the width of the specimen shall be the thickness of the sheet, and the depth shall not exceed the width (see Notes 4 and 5). For all tests, the support span shall be 16 (tolerance ±1) times the depth of the beam. Specimen width shall not exceed one fourth of the support span for specimens greater than 3.2 mm [⅛ in.] in depth. Specimens 3.2 mm or less in depth shall be 12.7 mm [½ in.] in width. The specimen shall be long enough to allow for overhanging on each end of at least 10 % of the support span, but in no case less than 6.4 mm [¼ in.] on each end. Overhang shall be sufficient to prevent the specimen from slipping through the supports.

NOTE 4—Whenever possible, the original surface of the sheet shall be unaltered. However, where testing machine limitations make it impossible to follow the above criterion on the unaltered sheet, one or both surfaces shall be machined to provide the desired dimensions, and the location of the specimens with reference to the total depth shall be noted. The value obtained on specimens with machined surfaces may differ from those obtained on specimens with original surfaces. Consequently, any specifications for flexural properties on thicker sheets must state whether the original surfaces are to be retained or not. When only one surface was machined, it must be stated whether the machined surface was on the tension or compression side of the beam.

NOTE 5—Edgewise tests are not applicable for sheets that are so thin that specimens meeting these requirements cannot be cut. If specimen depth exceeds the width, buckling may occur.

7.2.2 *Materials Less than 1.6 mm [1⁄16 in.] in Thickness*—The specimen shall be 50.8 mm [2 in.] long by 12.7 mm [½ in.] wide, tested flatwise on a 25.4-mm [1-in.] support span.

NOTE 6—Use of the formulas for simple beams cited in these test methods for calculating results presumes that beam width is small in comparison with the support span. Therefore, the formulas do not apply rigorously to these dimensions.

NOTE 7—Where machine sensitivity is such that specimens of these dimensions cannot be measured, wider specimens or shorter support spans, or both, may be used, provided the support span-to-depth ratio is at least 14 to 1. All dimensions must be stated in the report (see also Note 6).

7.3 *Laminated Thermosetting Materials and Sheet and Plate Materials Used for Electrical Insulation, Including Vulcanized Fiber and Glass-Bonded Mica*—For paper-base and fabric-base grades over 25.4 mm [1 in.] in nominal thickness, the specimens shall be machined on both surfaces to a depth of 25.4 mm. For glass-base and nylon-base grades, specimens over 12.7 mm [½ in.] in nominal depth shall be machined on both surfaces to a depth of 12.7 mm. The support span-to-depth ratio shall be chosen such that failures occur in the outer fibers of the specimens, due only to the bending moment (see Note 8). Therefore, a ratio larger than 16:1 may be necessary (32:1 or 40:1 are recommended). When laminated materials exhibit low compressive strength perpendicular to the laminations, they shall be loaded with a large radius loading nose (up to four times the specimen depth to prevent premature damage to the outer fibers.

7.4 *Molding Materials (Thermoplastics and Thermosets)*—The recommended specimen for molding materials is 127 by 12.7 by 3.2 mm [5 by ½ by ⅛ in.] tested flatwise on a support span, resulting in a support span-to-depth ratio of 16 (tolerance ±1). Thicker specimens should be avoided if they exhibit significant shrink marks or bubbles when molded.

7.5 *High-Strength Reinforced Composites, Including Highly Orthotropic Laminates*—The span-to-depth ratio shall be chosen such that failure occurs in the outer fibers of the specimens and is due only to the bending moment (see Note 8). A span-to-depth ratio larger than 16:1 may be necessary (32:1 or 40:1 are recommended). For some highly anisotropic composites, shear deformation can significantly influence modulus measurements, even at span-to-depth ratios as high as 40:1. Hence, for these materials, an increase in the span-to-depth ratio to 60:1 is recommended to eliminate shear effects when modulus data are required, it should also be noted that the flexural modulus of highly anisotropic laminates is a strong function of ply-stacking sequence and will not necessarily correlate with tensile modulus, which is not stacking-sequence dependent.

NOTE 8—As a general rule, support span-to-depth ratios of 16:1 are satisfactory when the ratio of the tensile strength to shear strength is less than 8 to 1, but the support span-to-depth ratio must be increased for composite laminates having relatively low shear strength in the plane of the laminate and relatively high tensile strength parallel to the support span.

8. Number of Test Specimens

8.1 Test at least five specimens for each sample in the case of isotropic materials or molded specimens.

8.2 For each sample of anisotropic material in sheet form, test at least five specimens for each of the following conditions. Recommended conditions are flatwise and edgewise tests on specimens cut in lengthwise and crosswise directions of the sheet. For the purposes of this test, “lengthwise” designates the principal axis of anisotropy and shall be interpreted to mean the direction of the sheet known to be stronger in flexure. “Crosswise” indicates the sheet direction known to be the weaker in flexure and shall be at 90° to the lengthwise direction.

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 unless otherwise specified by contract or the relevant ASTM material specification. Reference pre-test conditioning, to settle disagreements, shall apply tolerances of ±1°C [1.8°F] and ±2 % relative humidity.

9.2 *Test Conditions*—Conduct the tests at 23 ± 2°C [73.4 ± 3.6°F] and 50 ± 5 % relative humidity unless otherwise specified by contract or the relevant ASTM material specification. Reference testing conditions, to settle disagreements, shall apply tolerances of ±1°C [1.8°F] and ±2 % relative humidity.

10. Procedure

10.1 *Procedure A*:

10.1.1 Use an untested specimen for each measurement. Measure the width and depth of the specimen to the nearest 0.03 mm [0.001 in.] at the center of the support span. For specimens less than 2.54 mm [0.100 in.] in depth, measure the depth to the nearest 0.003 mm [0.0005 in.]. These measurements shall be made in accordance with Test Methods D 5947.

10.1.2 Determine the support span to be used as described in Section 7 and set the support span to within 1 % of the determined value.

10.1.3 For flexural fixtures that have continuously adjustable spans, measure the span accurately to the nearest 0.1 mm [0.004 in.] for spans less than 63 mm [2.5 in.] and to the nearest 0.3 mm [0.012 in.] for spans greater than or equal to 63 mm [2.5 in.]. Use the actual measured span for all calculations. For flexural fixtures that have fixed machined span positions, verify the span distance the same as for adjustable spans at each machined position. This distance becomes the span for that position and is used for calculations applicable to all subsequent tests conducted at that position. See Annex A2 for information on the determination of and setting of the span.

10.1.4 Calculate the rate of crosshead motion as follows and set the machine for the rate of crosshead motion as calculated by Eq 1:

$$R = ZL^2/6d \qquad (1)$$

where:
R = rate of crosshead motion, mm [in.]/min,
L = support span, mm [in.],
d = depth of beam, mm [in.], and
Z = rate of straining of the outer fiber, mm/mm/min [in./in./min]. Z shall be equal to 0.01.

In no case shall the actual crosshead rate differ from that calculated using Eq 1, by more than ±10 %.

10.1.5 Align the loading nose and supports so that the axes of the cylindrical surfaces are parallel and the loading nose is midway between the supports. The parallelism of the apparatus may be checked by means of a plate with parallel grooves into which the loading nose and supports will fit when properly aligned (see A2.3). Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading nose and supports.

10.1.6 Apply the load to the specimen at the specified crosshead rate, and take simultaneous load-deflection data. Measure deflection either by a gage under the specimen in contact with it at the center of the support span, the gage being mounted stationary relative to the specimen supports, or by measurement of the motion of the loading nose relative to the supports. Load-deflection curves may be plotted to determine the flexural strength, chord or secant modulus or the tangent modulus of elasticity, and the total work as measured by the area under the load-deflection curve. Perform the necessary toe compensation (see Annex A1) to correct for seating and indentation of the specimen and deflections in the machine.

10.1.7 Terminate the test when the maximum strain in the outer surface of the test specimen has reached 0.05 mm/mm [in./in.] or at break if break occurs prior to reaching the maximum strain (Notes 9 and 10). The deflection at which this strain will occur may be calculated by letting r equal 0.05 mm/mm [in./in.] in Eq 2:

$$D = rL^2/6d \qquad (2)$$

where:
D = midspan deflection, mm [in.],
r = strain, mm/mm [in./in.],
L = support span, mm [in.], and
d = depth of beam, mm [in.].

NOTE 9—For some materials that do not yield or break within the 5 % strain limit when tested by Procedure A, the increased strain rate allowed by Procedure B (see 10.2) may induce the specimen to yield or break, or both, within the required 5 % strain limit.

NOTE 10—Beyond 5 % strain, this test method is not applicable. Some other mechanical property might be more relevant to characterize materials that neither yield nor break by either Procedure A or Procedure B within the 5 % strain limit (for example, Test Method D 638 may be considered).

10.2 *Procedure B*:

10.2.1 Use an untested specimen for each measurement.

10.2.2 Test conditions shall be identical to those described in 10.1, except that the rate of straining of the outer surface of the test specimen shall be 0.10 mm/mm [in./in.]/min.

10.2.3 If no break has occurred in the specimen by the time the maximum strain in the outer surface of the test specimen has reached 0.05 mm/mm [in./in.], discontinue the test (see Note 10).

11. Retests

11.1 Values for properties at rupture shall not be calculated for any specimen that breaks at some obvious, fortuitous flaw, unless such flaws constitute a variable being studied. Retests shall be made for any specimen on which values are not calculated.

12. Calculation

12.1 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.

12.2 *Flexural Stress* (σ_f)—When a homogeneous elastic material is tested in flexure as a simple beam supported at two points and loaded at the midpoint, the maximum stress in the outer surface of the test specimen occurs at the midpoint. This stress may be calculated for any point on the load-deflection curve by means of the following equation (see Notes 11-13):

$$\sigma_f = 3PL/2bd^2 \qquad (3)$$

where:
σ = stress in the outer fibers at midpoint, MPa [psi],

P = load at a given point on the load-deflection curve, N [lbf],
L = support span, mm [in.],
b = width of beam tested, mm [in.], and
d = depth of beam tested, mm [in.].

NOTE 11—Eq 3 applies strictly to materials for which stress is linearly proportional to strain up to the point of rupture and for which the strains are small. Since this is not always the case, a slight error will be introduced if Eq 3 is used to calculate stress for materials that are not true Hookean materials. The equation is valid for obtaining comparison data and for specification purposes, but only up to a maximum fiber strain of 5 % in the outer surface of the test specimen for specimens tested by the procedures described herein.

NOTE 12—When testing highly orthotropic laminates, the maximum stress may not always occur in the outer surface of the test specimen.[7] Laminated beam theory must be applied to determine the maximum tensile stress at failure. If Eq 3 is used to calculate stress, it will yield an apparent strength based on homogeneous beam theory. This apparent strength is highly dependent on the ply-stacking sequence of highly orthotropic laminates.

NOTE 13—The preceding calculation is not valid if the specimen slips excessively between the supports.

12.3 *Flexural Stress for Beams Tested at Large Support Spans* (σ_f)—If support span-to-depth ratios greater than 16 to 1 are used such that deflections in excess of 10 % of the support span occur, the stress in the outer surface of the specimen for a simple beam can be reasonably approximated with the following equation (see Note 14):

$$\sigma_f = (3PL/2bd^2)[1 + 6(D/L)^2 - 4(d/L)(D/L)] \tag{4}$$

where:

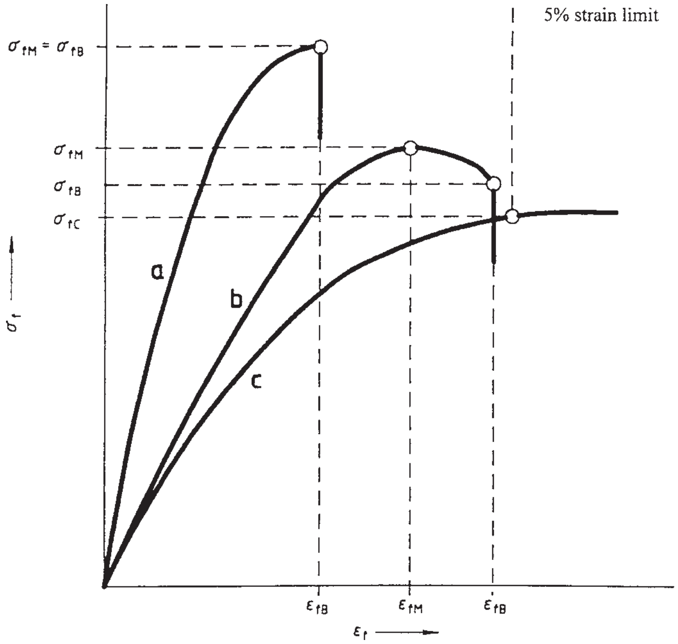
σ_f, P, L, b, and d are the same as for Eq 3, and
D = deflection of the centerline of the specimen at the middle of the support span, mm [in.].

NOTE 14—When large support span-to-depth ratios are used, significant end forces are developed at the support noses which will affect the moment in a simple supported beam. Eq 4 includes additional terms that are an approximate correction factor for the influence of these end forces in large support span-to-depth ratio beams where relatively large deflections exist.

12.4 *Flexural Strength* (σ_{fM})—Maximum flexural stress sustained by the test specimen (see Note 12) during a bending test. It is calculated according to Eq 3 or Eq 4. Some materials that do not break at strains of up to 5 % may give a load deflection curve that shows a point at which the load does not increase with an increase in strain, that is, a yield point (Fig. 2, Curve B), *Y*. The flexural strength may be calculated for these materials by letting *P* (in Eq 3 or Eq 4) equal this point, *Y*.

12.5 *Flexural Offset Yield Strength*—Offset yield strength is the stress at which the stress-strain curve deviates by a given strain (offset) from the tangent to the initial straight line portion of the stress-strain curve. The value of the offset must be given whenever this property is calculated.

NOTE 15—This value may differ from flexural strength defined in 12.4.



NOTE—Curve a: Specimen that breaks before yielding.
Curve b: Specimen that yields and then breaks before the 5 % strain limit.
Curve c: Specimen that neither yields nor breaks before the 5 % strain limit.

FIG. 2 Typical Curves of Flexural Stress (σ_f) Versus Flexural Strain (ϵ_f)

Both methods of calculation are described in the annex to Test Method D 638.

12.6 *Flexural Stress at Break* (σ_{fB})—Flexural stress at break of the test specimen during a bending test. It is calculated according to Eq 3 or Eq 4. Some materials may give a load deflection curve that shows a break point, *B*, without a yield point (Fig. 2, Curve a) in which case $\sigma_{fB} = \sigma_{fM}$. Other materials may give a yield deflection curve with both a yield and a break point, *B* (Fig. 2, Curve b). The flexural stress at break may be calculated for these materials by letting *P* (in Eq 3 or Eq 4) equal this point, *B*.

12.7 *Stress at a Given Strain*—The stress in the outer surface of a test specimen at a given strain may be calculated in accordance with Eq 3 or Eq 4 by letting *P* equal the load read from the load-deflection curve at the deflection corresponding to the desired strain (for highly orthotropic laminates, see Note 12).

12.8 *Flexural Strain*, ϵ_f—Nominal fractional change in the length of an element of the outer surface of the test specimen at midspan, where the maximum strain occurs. It may be calculated for any deflection using Eq 5:

$$\epsilon_f = 6Dd/L^2 \tag{5}$$

where:
ϵ_f = strain in the outer surface, mm/mm [in./in.],
D = maximum deflection of the center of the beam, mm [in.],
L = support span, mm [in.], and

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[7] For a discussion of these effects, see Zweben, C., Smith, W. S., and Wardle, M. W., "Test Methods for Fiber Tensile Strength, Composite Flexural Modulus and Properties of Fabric-Reinforced Laminates, " *Composite Materials: Testing and Design (Fifth Conference), ASTM STP 674*, 1979, pp. 228–262.

d = depth, mm [in.].
D = maximum deflection of the center of the beam, mm [in.],
L = support span, mm [in.], and
d = depth, mm [in.].

12.9 *Modulus of Elasticity*:

12.9.1 *Tangent Modulus of Elasticity*—The tangent modulus of elasticity, often called the "modulus of elasticity," is the ratio, within the elastic limit, of stress to corresponding strain. It is calculated by drawing a tangent to the steepest initial straight-line portion of the load-deflection curve and using Eq 6 (for highly anisotropic composites, see Note 16).

$$E_B = L^3 m / 4bd^3 \tag{6}$$

where:
E_B = modulus of elasticity in bending, MPa [psi],
L = support span, mm [in.],
b = width of beam tested, mm [in.],
d = depth of beam tested, mm [in.], and
m = slope of the tangent to the initial straight-line portion of the load-deflection curve, N/mm [lbf/in.] of deflection.

NOTE 16—Shear deflections can seriously reduce the apparent modulus of highly anisotropic composites when they are tested at low span-to-depth ratios.[7] For this reason, a span-to-depth ratio of 60 to 1 is recommended for flexural modulus determinations on these composites. Flexural strength should be determined on a separate set of replicate specimens at a lower span-to-depth ratio that induces tensile failure in the outer fibers of the beam along its lower face. Since the flexural modulus of highly anisotropic laminates is a critical function of ply-stacking sequence, it will not necessarily correlate with tensile modulus, which is not stacking-sequence dependent.

12.9.2 *Secant Modulus*— The secant modulus is the ratio of stress to corresponding strain at any selected point on the stress-strain curve, that is, the slope of the straight line that joins the origin and a selected point on the actual stress-strain curve. It shall be expressed in megapascals [pounds per square inch]. The selected point is chosen at a prespecified stress or strain in accordance with the appropriate material specification or by customer contract. It is calculated in accordance with Eq 6 by letting m equal the slope of the secant to the load-deflection curve. The chosen stress or strain point used for the determination of the secant shall be reported.

12.9.3 *Chord Modulus* (E_f)—The chord modulus may be calculated from two discrete points on the load deflection curve. The selected points are to be chosen at two prespecified stress or strain points in accordance with the appropriate material specification or by customer contract. The chosen stress or strain points used for the determination of the chord modulus shall be reported. Calculate the chord modulus, E_f using the following equation:

$$E_f = (\sigma_{f2} - \sigma_{f1})/(\epsilon_{f2} - \epsilon_{f1}) \tag{7}$$

where:

σ_{f2} and σ_{f1} are the flexural stresses, calculated from Eq 3 or Eq 4 and measured at the predefined points on the load deflection curve, and ϵ_{f2} and

ϵ_{f1} are the flexural strain values, calculated from Eq 5 and measured at the predetermined points on the load deflection curve.

12.10 *Arithmetic Mean*— For each series of tests, the arithmetic mean of all values obtained shall be calculated to three significant figures and reported as the "average value" for the particular property in question.

12.11 *Standard Deviation*—The standard deviation (estimated) shall be calculated as follows and be reported to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\bar{X}^2)/(n-1)} \tag{8}$$

where:
s = estimated standard deviation,
X = value of single observation,
n = number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

13. Report

13.1 Report the following information:

13.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, and previous history (for laminated materials, ply-stacking sequence shall be reported),

13.1.2 Direction of cutting and loading specimens, when appropriate,

13.1.3 Conditioning procedure,

13.1.4 Depth and width of specimen,

13.1.5 Procedure used (A or B),

13.1.6 Support span length,

13.1.7 Support span-to-depth ratio if different than 16:1,

13.1.8 Radius of supports and loading noses if different than 5 mm,

13.1.9 Rate of crosshead motion,

13.1.10 Flexural strain at any given stress, average value and standard deviation,

13.1.11 If a specimen is rejected, reason(s) for rejection,

13.1.12 Tangent, secant, or chord modulus in bending, average value, standard deviation, and the strain level(s) used if secant or chord modulus,

13.1.13 Flexural strength (if desired), average value, and standard deviation,

TABLE 2 Flexural Modulus

Material	Mean, 10^3 psi	Values Expressed in units of % of 10^3 psi			
		V_r[A]	V_R[B]	r[C]	R[D]
ABS	338	4.79	7.69	13.6	21.8
DAP thermoset	485	2.89	7.18	8.15	20.4
Cast acrylic	810	13.7	16.1	38.8	45.4
GR polyester	816	3.49	4.20	9.91	11.9
GR polycarbonate	1790	5.52	5.52	15.6	15.6
SMC	1950	10.9	13.8	30.8	39.1

[A] V_r = within-laboratory coefficient of variation for the indicated material. It is obtained by first pooling the within-laboratory standard deviations of the test results from all of the participating laboratories: $Sr = [[(s_1)^2 + (s_2)^2 \ldots + (s_n)^2]/n]^{1/2}$ then $V_r = (S_r$ divided by the overall average for the material) × 100.

[B] V_r = between-laboratory reproducibility, expressed as the coefficient of variation: $S_R = \{S_r^2 + S_L^2\}^{1/2}$ where S_L is the standard deviation of laboratory means. Then: $V_R = (S_R$ divided by the overall average for the material) × 100.

[C] r = within-laboratory critical interval between two test results = 2.8 × V_r.

[D] R = between-laboratory critical interval between two test results = 2.8 × V_R.

13.1.14 Stress at any given strain up to and including 5 % (if desired), with strain used, average value, and standard deviation,

13.1.15 Flexural stress at break (if desired), average value, and standard deviation,

13.1.16 Type of behavior, whether yielding or rupture, or both, or other observations, occurring within the 5 % strain limit, and

13.1.17 Date of specific version of test used.

14. Precision and Bias [8]

14.1 Tables 1 and 2 are based on a round-robin test conducted in 1984, in accordance with Practice E 691, involving six materials tested by six laboratories using Procedure A. For each material, all the specimens were prepared at one source. Each "test result" was the average of five individual determinations. Each laboratory obtained two test results for each material.

NOTE 17—**Caution:** The following explanations of *r* and *R* (14.2-14.2.3) are intended only to present a meaningful way of considering the approximate precision of these test methods. The data given in Tables 2 and 3 should not be applied rigorously to the acceptance or rejection of materials, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of these test methods should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 14.2-14.2.3 would then be valid for such data.

14.2 *Concept of "r" and "R" in* Tables 1 and 2—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing five specimens for each test result, then:

14.2.1 *Repeatability*— Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material. *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.2.2 *Reproducibility*— Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the *R* value for that material. *R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.2.3 The judgments in 14.2.1 and 14.2.2 will have an approximately 95 % (0.95) probability of being correct.

14.3 *Bias*—No statement may be made about the bias of these test methods, as there is no standard reference material or reference test method that is applicable.

15. Keywords

15.1 flexural properties; plastics; stiffness; strength

ANNEXES

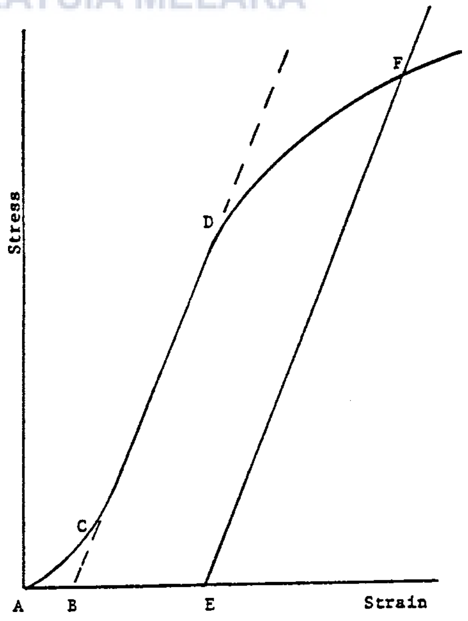
(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (see 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 (see 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 (see 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



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

FIG. A1.1 Material with Hookean Region

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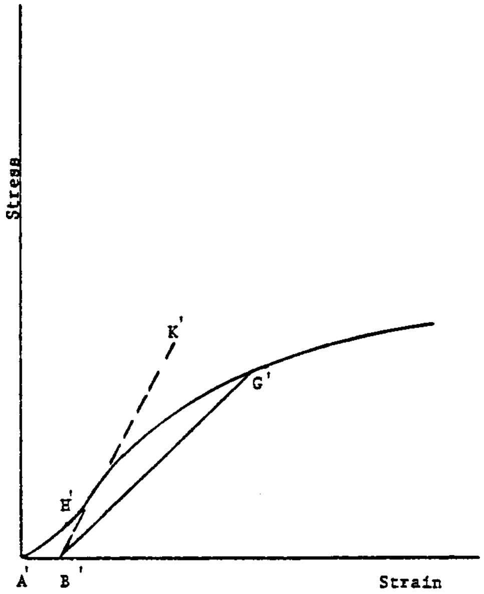
[8] Supporting data are available from ASTM Headquarters. Request RR: D20 – 1128.

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

FIG. A1.2 Material with No Hookean Region

A2. MEASURING AND SETTING SPAN

A2.1 For flexural fixtures that have adjustable spans, it is important that the span between the supports is maintained constant or the actual measured span is used in the calculation of stress, modulus, and strain, and the loading nose or noses are positioned and aligned properly with respect to the supports. Some simple steps as follows can improve the repeatability of your results when using these adjustable span fixtures.

A2.2 Measurement of Span:

A2.2.1 This technique is needed to ensure that the correct span, not an estimated span, is used in the calculation of results.

A2.2.2 Scribe a permanent line or mark at the exact center of the support where the specimen makes complete contact. The type of mark depends on whether the supports are fixed or rotatable (see Figs. A2.1 and A2.2).

A2.2.3 Using a vernier caliper with pointed tips that is readable to at least 0.1 mm [0.004 in.], measure the distance between the supports, and use this measurement of span in the calculations.

FIG. A2.1 Markings on Fixed Specimen Supports

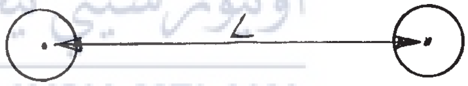


FIG. A2.2 Markings on Rotatable Specimen Supports

A2.3 *Setting the Span and Alignment of Loading Nose(s)*—To ensure a consistent day-to-day setup of the span and ensure the alignment and proper positioning of the loading nose, simple jigs should be manufactured for each of the standard setups used. An example of a jig found to be useful is shown in Fig. A2.3.

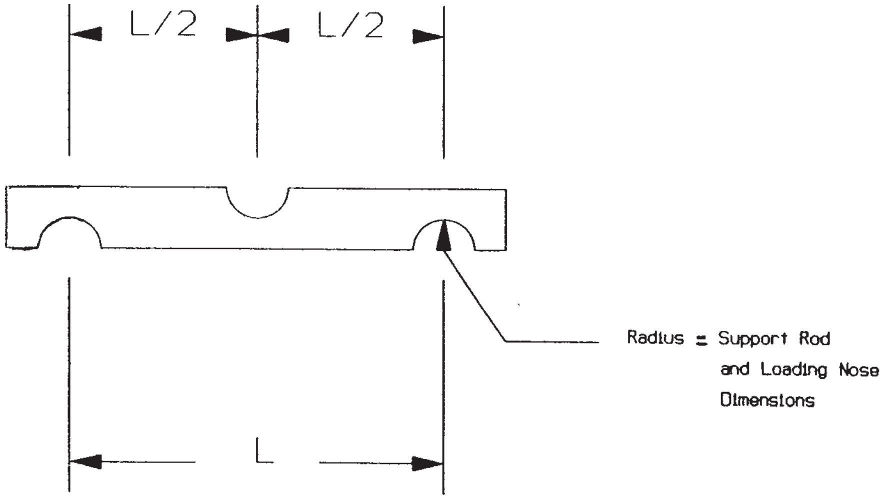


FIG. A2.3 Fixture Used to Set Loading Nose and Support Spacing and Alignment

APPENDIX

(Nonmandatory Information)

X1. DEVELOPMENT OF A FLEXURAL MACHINE COMPLIANCE CORRECTION

X1.1 Introduction

X1.1.1 Universal Testing instrument drive systems always exhibit a certain level of compliance that is characterized by a variance between the reported crosshead displacement and the displacement actually imparted to the specimen. This variance is a function of load frame stiffness, drive system wind-up, load cell compliance and fixture compliance. To accurately measure the flexural modulus of a material, this compliance should be measured and empirically subtracted from test data. Flexural modulus results without the corrections are lower than if the correction is applied. The greater the stiffness of the material the more influence the system compliance has on results.

X1.1.2 It is not necessary to make the machine compliance correction when a deflectometer/extensometer is used to measure the actual deflection occurring in the specimen as it is deflected.

X1.2 Terminology

X1.2.1 *Compliance*—The displacement difference between test machine drive system displacement values and actual specimen displacement

X1.2.2 *Compliance Correction*—An analytical method of modifying test instrument displacement values to eliminate the amount of that measurement attributed to test instrument compliance.

X1.3 Apparatus

X1.3.1 Universal Testing machine

X1.3.2 Load cell

X1.3.3 Flexure fixture including loading nose and specimen supports

X1.3.4 Computer Software to make corrections to the displacements

X1.3.5 Steel bar, with smoothed surfaces and a calculated flexural stiffness of more than 100 times greater than the test material. The length should be at least 13 mm greater than the support span. The width shall match the width of the test specimen and the thickness shall be that required to achieve or exceed the target stiffness.

X1.4 Safety Precautions

X1.4.1 The universal testing machine should stop the machine crosshead movement when the load reaches 90 % of load cell capacity, to prevent damage to the load cell.

X1.4.2 The compliance curve determination should be made at a speed no higher than 2 mm/min. Because the load builds up rapidly since the steel bar does not deflect, it is quite easy to exceed the load cell capacity.

X1.5 Procedure

NOTE X1.1—A new compliance correction curve should be established each time there is a change made to the setup of the test machine, such as, load cell changed or reinstallation of the flexure fixture on the machine. If the test machine is dedicated to flexural testing, and there are no changes to the setup, it is not necessary to re-calculate the compliance curve.

NOTE X1.2—On those machines with computer software that automatically make this compliance correction; refer to the software manual to determine how this correction should be made.

X1.5.1 The procedure to determine compliance follows:

X1.5.1.1 Configure the test system to match the actual test configuration.

X1.5.1.2 Place the steel bar in the test fixture, duplicating the position of a specimen during actual testing.

X1.5.1.3 Set the crosshead speed to 2 mm/min. or less and start the crosshead moving in the test direction recording crosshead displacement and the corresponding load values.

X1.5.1.4 Increase load to a point exceeding the highest load expected during specimen testing. Stop the crosshead and return to the pre-test location.

X1.5.1.5 The recorded load-deflection curve, starting when the loading nose contacts the steel bar to the time that the highest load expected is defined as test system compliance.

X1.5.2 Procedure to apply compliance correction is as follows:

X1.5.2.1 Run the flexural test method on the material at the crosshead required for the measurement.

X1.5.2.2 It is preferable that computer software be used to make the displacement corrections, but if it is not available compliance corrections can be made manually in the following manner. Determine the range of displacement (D) on the load versus displacement curve for the material, over which the modulus is to be calculated. For Young's Modulus that would steepest region of the curve below the proportional limit. For Secant and Chord Modulii that would be at specified level of strain or specified levels of strain, respectively. Draw two vertical lines up from the displacement axis for the two chosen displacements (D1, D2) to the load versus displacement curve for the material. In some cases one of these points maybe at zero displacement after the toe compensation correction is made. Draw two horizontal lines from these points on the load displacement curve to the Load (P) axis. Determine the loads (L1, L2).

X1.5.2.3 Using the Compliance Correction load displacement curve for the steel bar, mark off L1 and L2 on the Load (P) axis. From these two points draw horizontal lines across till they contact the load versus displacement curve for the steel bar. From these two points on the load deflection curve draw two vertical lines downwards to the displacement axis. These two points on the displacement axis determine the corrections (c1, c2) that need to be made to the displacements measurements for the test material.

X1.5.2.4 Subtract the corrections (c1, c2) from the measured displacements (D1, D2), so that a true measures of test specimen deflection (D1-c1, D2-c2) are obtained.

X1.6 Calculations

X1.6.1 Calculation of Chord Modulus

X1.6.1.1 Calculate the stresses ($\sigma f1$, $\sigma f2$) for load points L1 and L2 from Fig. X1.1 using the equation in 12.2 3.

X1.6.1.2 Calculate the strains ($\epsilon f1$, $\epsilon f2$) for displacements D1-c1 and D2-c2 from Fig. X1.3 using the equation in 12.8 Eq. 5.

X1.6.1.3 Calculate the flexural chord modulus in accordance with 12.9.3 Eq. 7.

X1.6.2 Calculation of Secant Modulus

X1.6.2.1 Calculation of the Secant Modulus at any strain along the curve would be the same as conducting a chord modulus measurement, except that $\sigma f1 = 0$, L1= 0, and D1-c1 = 0.

X1.6.3 Calculation of Young's Modulus

X1.6.3.1 Determine the steepest slope "m" along the curve, below the proportional limit, using the selected loads L1 and L2 from Fig. X1.1 and the displacements D1-c1 and D2-c2 from Fig. X1.3.

X1.6.3.2 Calculate the Young's modulus in accordance with 12.9.1 Eq. 6.

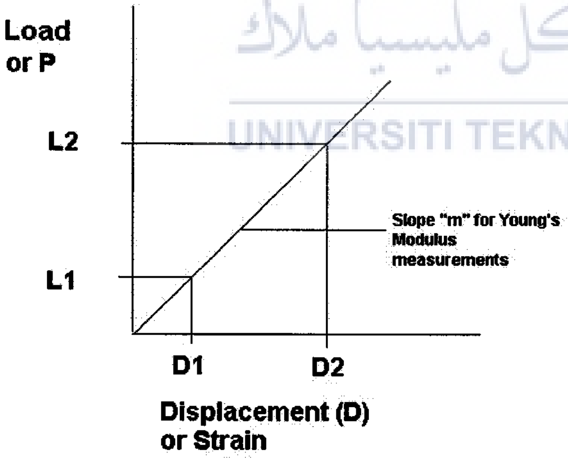


FIG. X1.1 Example of Modulus Curve for a Material

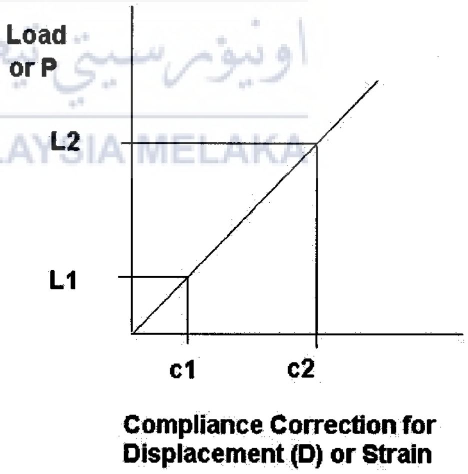


FIG. X1.2 Compliance Curve for Steel Bar

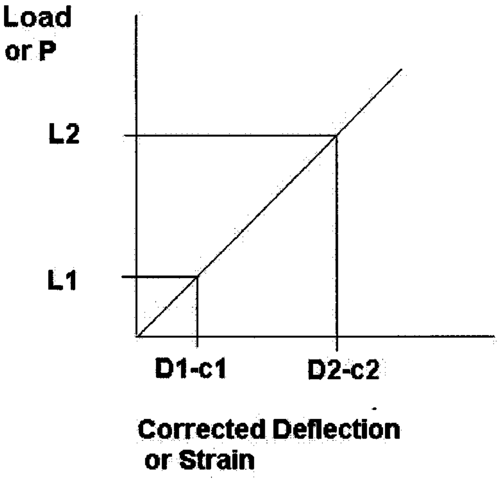


FIG. X1.3 Example of the Material Curve Corrected for the Compliance Corrected Displacement or Strain

SUMMARY OF CHANGES

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

D 790 – 03:

(*1*) Added Appendix X1.

D 790 – 02:

(*1*) Revised 9.1 and 9.2.

D 790 – 00:

(*1*) Revised 12.1.

D 790 – 99:

(*1*) Revised 10.1.3.

D 790 – 98:

(*1*) Section 4.2 was rewritten extensively to bring this standard closer to ISO 178.

(*2*) Fig. 2 was added to clarify flexural behaviors that may be observed and to define what yielding and breaking behaviors look like, as well as the appropriate place to select these points on the stress strain curve.

Designation: C 559 – 90 (Reapproved 2005)

An American National Standard

Standard Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles[1]

This standard is issued under the fixed designation C 559; 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.

1. Scope

1.1 This test method covers the determination of the bulk density of manufactured articles of carbon and graphite of at least 500 mm^3 volume. The bulk density is calculated to an accuracy of 0.25 %, using measurements of mass and dimensions in air at 25 ± 5°C.

1.2 *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:* [2]

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI) (the Modern Metric System)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *bulk density* —the mass of a unit volume of material including both permeable and impermeable voids.

4. Significance and Use

4.1 Bulk density as determined by this test method is a basic material property of importance in manufacturing and application of carbon and graphite.

4.2 This test method can be used for quality and process control, material characterization and description, and other purposes.

5. Preparation of Test Specimens

5.1 Machine test specimens from the manufactured article in the form of a rectangular parallelepiped or a right circular cylinder. The minimum mass of the specimen shall be 2000 times the sensitivity of the balance used to weigh the specimen, and the volume of the specimen shall not be less than 500 mm^3. The minimum dimension of the specimen shall be the larger of:

5.1.1 Ten times the length of the largest visible particle, and

5.1.2 2000 times the resolution of the device used for measuring the dimension.

5.2 During the machining operation, use no lubricant having a boiling point above 100°C. All corners, edges, and faces of the specimen should be free of chips or gouges. Ensure that the specimen is free of any residue from the machining operation. Dry the specimen for a minimum of 2 h at 110°C, and then allow it to cool to 25 ± 5°C in a desiccator. The specimen shall not be removed from the desiccator until immediately prior to weighing.

6. Procedure

6.1 Weigh the specimen to an accuracy of 0.05 % using a balance or scale. During the weighing operation, handle the specimen with soft-tipped tongs.

6.2 Measure each dimension of the test specimen to an accuracy of 0.05 %.

6.2.1 If the specimen is a rectangular parallelepiped, make four measurements of the length (longest dimension). Take each measurement along the center of each of the four long faces of the specimen. Measure the width and thickness at each end and at two intermediate points along the length of the specimen. Determine the mean of each dimension.

6.2.2 If the specimen is a right circular cylinder, measure the length at four points, 90° apart on the periphery of the circular end faces. Make two sets of diameter measurements. Each set shall consist of four measurements, one at each end and two at intermediate points along an axial line. These sets shall lie at 90° to each other. Determine the mean length and the mean of each of the two sets of diameter measurements.

7. Calculation

7.1 Convert the mass to milligrams and the dimensions to millimetres, or convert the mass to megagrams and the dimensions to metres. Conversion factors are available in IEEE/ASTM SI-10.

7.2 The volume of the specimen may be calculated as follows:

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

Current edition approved June 1, 2005. Published August 2005. Originally approved in 1968. Last previous edition approved in 2000 as C 559–90(2000).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

For a rectangular parallelepiped:

$$V = l\,wt \tag{1}$$

where:

V = volume, mm^3(or m^3),
l = mean length, mm (or m),
w = mean width, mm (or m), and
t = mean thickness, mm (or m).

For a right circular cylinder:

$$V = (\pi d_1 d_2 l)/4 \tag{2}$$

where:

d_1, d_2 = mean diameters, mm (or m).

7.3 The bulk density of the specimen may be calculated as follows:

$$D = M/V \tag{3}$$

where:

D = bulk density, mg/mm^3(or Mg/m^3),
M = mass, mg (or Mg), and
V = volume, mm^3(or m^3).

8. Report

8.1 Report the following:

8.1.1 Type, source, grade, and form of the sample, and

8.1.2 Densities of the individual specimens and the mean.

9. Precision and Bias

9.1 No numerical statement can be made on the intralaboratory or interlaboratory precision of this test method, since such tests have not been run. The resultant density will be accurate to within the claimed 0.25 % if the following conditions are met in their entirety:

9.1.1 All measuring devices (calipers, scales, and balances) have accuracy, at the time of use, equal to the resolution of the device. Such accuracy can be verified by measurement of standards before and after any series of density determinations, plus a program of regular calibration of standards.

9.1.2 The machined specimens have right angles accurate to ±1°.

9.1.3 The machined specimens have plane surfaces flat to within 0.05 % of the dimension perpendicular to the plane. If the specimen is a rectangular parallelepiped, nonparallel opposite sides will cause a systematic error if a standard micrometer having flat anvil faces is used. (If a round anvil micrometer is used, disregard the following.) The systematic error will be greater than the accuracy of the measurement if the measurements of a given dimension are uniformly increasing or decreasing along the specimen, and if $\tan \theta > (0.001)$ (mean dimension being measured)/(micrometer anvil diameter), where θ is the angle by which the sides deviate from parallelism ($\theta = 0°$ for parallel sides). If $\tan \theta$ is greater than the specified tolerance, the specimen should be discarded.

9.2 If specimens having volumes close to the minimum (500 mm^3) are used, extra care should be taken to ensure that the specified accuracies are achieved.

9.3 Surface roughness may cause systematic errors in dimension measurements since micrometer calipers generally read surface peaks. In order to ensure that the accuracy standards for dimension measurements are met, the maximum peak-to-valley distance shall be less than 0.05 % of the dimension being measured.

9.4 The buoyant effect of the air will cause a systematic error. For typical carbon and graphite, the densities will be low by approximately 0.05 %. This systematic error has been taken into account in the overall bias (0.25 %) of the test method.

9.5 Errors can be introduced by deformation of specimens from application of force during dimensional measurement. Care must be taken not to exert force on a specimen during measurement, particularly when measuring a low modulus material.

9.6 Contamination of specimens during handling can cause the apparent mass to deviate from the true mass. The cautions in Sections 5 and 6 concerning specimen handling and cleanliness must be observed.

10. Keywords

10.1 bulk; carbon density; graphite; gravimetric; physical

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An American National Standard

Standard Test Method for Scleroscope Hardness Testing of Carbon and Graphite Materials[1]

This standard is issued under the fixed designation C 886; 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.

1. Scope

1.1 This test method covers the apparatus and procedure for determining the hardness of carbon and graphite materials using the Model C-2 scleroscope[2] with the hammer calibrated for use on carbon and graphite materials with particles smaller than 0.8 mm (0.032 in.).[3]

1.2 The values given in acceptable metric units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

1.3 *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:* [4]

E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials

E 448 Practice for Scleroscope Hardness Testing of Metallic Materials

2.2 *ANSI Standard:*[5]

ANSI C 64.1 Brushes for Electrical Machines

3. Summary of Test Method

3.1 The specimen is held in position, and the height of rebound of a diamond-tipped hammer is observed and recorded as the hardness number.

4. Significance and Use

4.1 The scleroscope is a rebound hardness tester with a scale divided into 140 equal parts. For carbon and graphite materials, there is no established correlation between the Scleroscope hardness scale and other hardness scales. The test is useful in the evaluation and the manufacturing control of carbon and graphite materials.

5. Interferences

5.1 Lack of alignment of the instrument, as specified in Section 10, will cause low readings.

5.2 The specimen must be held firmly in position and must have adequate support from the anvil on which the sample rests. Neglect of alignment, positioning, or support will result in low readings.

5.3 Rough surface finish, above 3175-nm (125-µin.) AA, may cause low readings.

5.4 Indentations that are superimposed or spaced too closely together (approximately 3 mm (⅛ in.)) will cause incorrect readings.

6. Apparatus

6.1 *Table*, or equivalent, firm, for support.

6.2 *Scleroscope*, Model C-2, equipped with a hammer calibrated by the manufacturer for use on carbon and graphite materials.

7. Test Specimen

7.1 The specimen may be of any convenient size, but test surfaces smaller than 5 by 5 mm (0.2 by 0.2 in.) are not recommended.

7.2 The recommended specimen shall have a minimum thickness of 5 mm (0.2 in.).

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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[2] Registered trademark of the Shore Instrument and Manufacturing Co., Inc., 80-A Commercial St., Freeport, NY 11520.

[3] This test method may be more readily understood by referring to the following documents: Practice E 448, ANSI C 64.1, Brushes for Electrical Machines, available from American National Standards Institute, 1430 Broadway, New York, NY 10017, and Lysaght and DeBellis, *Indentation Hardness Testing*, American Chain and Cable Corp., Reinhold Publishing Co., 1969.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

7.3 The test surface shall not exceed 3175-nm (125-µin.) AA in surface finish.

7.4 The test surface-to-opposite side parallelism shall be within 1 % (0.010 % per linear inch). For all specimens, the test surfaces must be maintained normal to the axis of the scleroscope tube.

8. Calibration and Standardization

8.1 Check the calibration of the Scleroscope with carbon test blocks obtained from the instrument manufacturer.

8.2 Make a series of tests on carbon test blocks that cover the range of hardness to be tested.

8.3 Make at least five tests on each carbon test block. Do not make any tests closer than 6 mm (0.25 in.) from the sides of the test blocks.

8.4 Consider the instrument calibrated if 90 % of the readings deviate no more than ±4 %, or ±3 points, whichever is larger, from the mean of the hardness numbers stamped on the carbon test blocks.

9. Conditioning

9.1 Allow the specimen to reach equilibrium with room temperature air, and test under the same conditions in accordance with Specification E 171.

9.2 Keep specimens free of contamination.

10. Procedure

10.1 Place the instrument on a firm support.

10.2 Level the instrument using the plumb bob or bubble level.

10.3 Check the calibration in accordance with Section 8.

10.4 Place the specimen on the anvil.

10.5 Be certain that the hammer is in the "UP'' (locked) position.

10.6 Lower the tube against the specimen, and hold firmly.

10.7 Release the hammer and read the height of the rebound.

10.8 Return the hammer to the "UP'' (locked) position.

10.9 Repeat 10.6 through 10.8 four or more times, using a different impact point each time in accordance with 8.4.

10.10 Record the average reading to the nearest whole number.

10.11 A single reading substantially different from the other shall be reported, but not included in the average or standard deviation calculations.

11. Report

11.1 Report the following information:

11.1.1 Material identification,

11.1.2 Specimen dimensions,

11.1.3 Surface conditions, that is, as formed or machined, wet or dry,

11.1.4 Orientation of the test surface with respect to anisotropy of material (parallel or perpendicular),

11.1.5 Number of readings taken,

11.1.6 Average hardness reading to the nearest whole number,

11.1.7 Standard deviation where applicable,

11.1.8 Testing conditions, including test date, serial number of test machine, and operator's name, and

11.1.9 Special environmental conditions, if any.

12. Precision and Bias [6]

12.1 Precision among six laboratories is ±3 points.

12.2 Bias of a properly calibrated instrument is ±4 %, or ±3 points, whichever is larger.

13. Keywords

13.1 carbon; graphite; hardness; Scleroscope

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C05–1005.

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