Microstructural Characterization Of Polymer Plate Washer Produce By A Novel Technique

by

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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

June 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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AZLIZA BINTI ISHAK

ABSTRACT

The overall objective from this project is to study the microstructure of the plate polymer washer produced by a new technique. The melt-shearing influenced the size of the spherulites and the formation of shish kebab structure.

From scanning electron microscopy (SEM), it revealed that for almost all polymers spherulites are composed of lamellar structure. The temperature will effect on the size and orientation of the polymer chains thus increases the toughness of the polymer. Melt-shearing also caused development of row-nuclei in the form of microfibrillar bundles and promoted the epitaxial growth of folded chain lamellae that filled the space normal (perpendicular) to the row-nuclei, resulting in a supramolecular structure of cylindrical symmetry or cylindrites. The rate of crystallization is profoundly altered by the influence of flow due to the alignment or orientation of molecular chains. From the nucleation sites oriented lamellae grow radially perpendicular to the shear flow direction. The developments of oriented morphologies in bulk polymers have been termed the ‘shish kebab’ model which the shish are the portions of oriented molecules and kebabs are the outgrowing lamellae.

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CHAPTER 1

INTRODUCTION

1.1 Background Study

1.1.1 Polymer Washer (hardware) [1]

Most tools utilized in the manufactory industrial use a washer to prevent the failure of the component. Washer is a thin plate typically in the disc shape with a hole that is normally used to distribute the load of a threaded fastener or hinge. Washers are used as a spacer, spring (belleville washer, wave washer), wear pad, preload indicating device, locking device and to reduce vibration. Washers usually have an outer diameter (OD) about twice the length of their inner diameter (ID). It was made usually from metal or plastic as shown in Figure 1 below. High quality caps crews require hardened steel washers to prevent the loss of pre-load due to Brinelling after the torque is applied. However, rubber or fiber gaskets used in taps (or faucets, or valves) to stop the flow of water are sometimes referred to colloquially as *washers*; but, while they may look similar, washers and gaskets are usually designed for different functions and made differently. [1]

The most common material to produce the polymer washers is rubber. Rubber washers may use for preventing galvanic corrosion, particularly by insulating steel screws from aluminum surfaces. Rubber washers also used in order to reduce vibration when

fastening fans to computer cases. Large door or gates always required polymer or rubber washers to prevent the friction when the door is open or close. Washers are made from the polymer have a smooth surface compared to steel washer. Washers are also important for preventing galvanic corrosion, particularly by insulating steel screws from aluminum surfaces. There are several methods to produce the polymer washer such as piercing, milling, casting and punching process. [1]

(a) (b)

Figure 1: Washer can be produce in various shape and size. (a) Metal washer (b) Rubber or polymer washer. [1]

1.1.2 Natural and Synthetic Polymer [2,15]

Polymer is a large molecule (macromolecule) composed of repeating structural units typically connected by covalent chemical bonds to form really long chains. Polymer in popular usage such as plastic, the term actually refers to a large class of natural and synthetic materials with a wide variety of properties. Natural polymeric materials consist of shellac, amber, and natural rubber have been in use for centuries. Biopolymers such as proteins and nucleic acids play crucial roles in biological processes. A variety of other natural polymers exist, such as cellulose, which is the main constituent of wood and paper. While the list of synthetic polymers includes synthetic rubber, Bakelite, neoprene, nylon, PVC, polystyrene, polyethylene, polypropylene, poly acrylonitrile, PVB and silicone.

1.1.3 Shearing process to produce polymer washer [3,19]

Shearing operations using a press tool including piercing, blanking, cropping and reducing burrs. The function of the processes is similar to each other which are to cut the metal or to make a hole of the materials. [3]

a) SHEARING PROCESS [3,19]

Operations that cut sheet metal using press tools in the shearing process as shown in Figure 2 below.

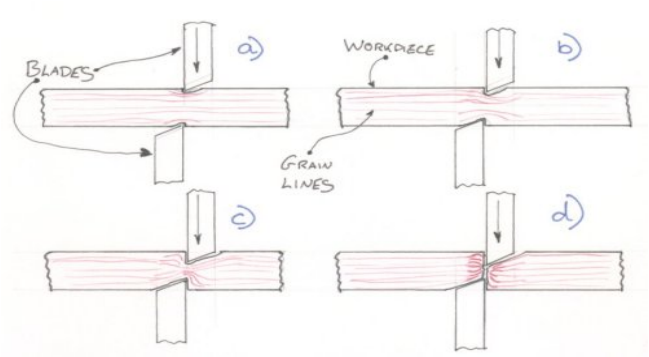


Figure 2: Four stages of the shearing process [3]

a. The blades begin to apply pressure; the workpiece starts to undergo elastic deformation.
b. As pressure increases the workpiece undergoes plastic deformation.
c. The blades begin to penetrate either side. Work hardening occurs in the centre.
d. Fracture lines originating from the point of each blade meet in the centre of the workpiece causing separation before the blades have fully penetrated.

To reduce the presence of burrs it is important that the blades are sharp. Also it is important that they do not deflect under pressure and that they are not set too far apart (offset), if this happens the fracture lines may not meet cleanly and the material will be dragged down the gap between the blades. Both effects cause excessive burrs and sharpness on the finished part.

b) PIERCING/PUNCHING [3,19]

Punching or piercing as shown in Figure 3 below is the common process to produce the plate polymer washer by using a punch press to push a punch through the material and into a die to create a hole in the washer. A punch is often made of hardened steel or carbides. The punch press forces the punch into a plate polymer piercing a hole that has a diameter equivalent to the punch. A die is located on the opposite side of the plate and supports the edge of the hole created to keep it from deforming during the punch.

There is a small amount of clearance between the punch's diameter and the die's. The slug from the hole falls through the die into some sort of container to either dispose of the slug or recycle it.

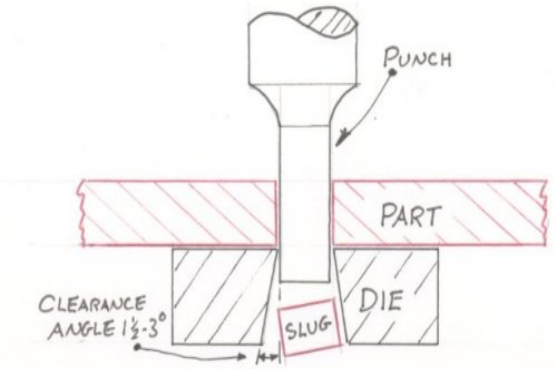


Figure 3: Piercing or Punching process [3]

The punching process causes an additional deformation. This deformation can be relatively large, which in case a dense pattern of holes is made or unfavourable process parameters are chosen. The deformations are a spring-back phenomenon and are caused by:

- Residual stresses already present the change in stiffness of the plate may result in a different equilibrium shape.
- Residual stresses near the cut-edge due to the separation process.

However, small plastic deformations in the area around the cut-edge and which occur when the blank holder force is too small can be of importance too. The deformations after cutting not only cause shape aberrations but also severe problems in subsequent manufacturing steps, such as poor fittability in joining processes. If the aberrations can be predicted, the product tolerances could be improved by adjusting the product design or manufacturing process. [3]

c) BLANKING [3,19]

Blanking is the same process as piercing which both process use a punch and die, but in this case it is the slug that is required. Blanked parts usually come from strip material fed through the press tool between guides. Furthermore, large blank area required a very high force which use of a powerful press machine. In order to improve productivity, it is quite normal to combine a piercing and blanking operation in one press tool, Figure 4 shows a typical follow on pierce and blank press tool to produce a plain washer.

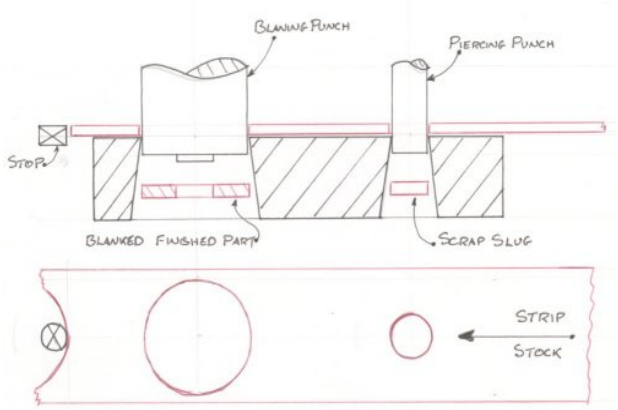


Figure 4: Blanking process [2, 3]

After each action of the press the strip is fed forward to stop against the previously blanked hole. The pilot on the blanking punch locates in the hole already pierced, prior to blanking the part from the strip.

d) CROPPING [3,19]

Cropping is the term applied when shearing across the full width of a strip. The cropping tool may be of any shape and produce a part of any length as shown in Figure 5 as below.

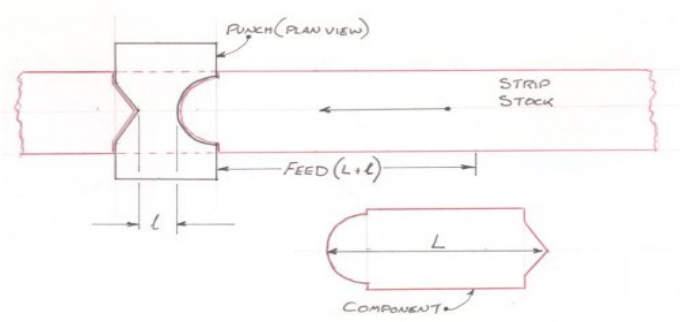


Figure 5: Cropping process [3]

1.2 Problem Statement

In washer production, shearing operation is always been used by the manufacturer to cut the materials into the desired shape. Shearing operation can be categorized into several processes which are shearing process, cropping, blanking, piercing and punching [2]. The shearing operation is the simplest and cheapest methods to produce the washer. However, shearing operation always contributes to the wasteful of material and it gives a bad impact on the microstructure of the material. As the punch uses the force to make the hole, sample such as a polymer might be shear thus it will disturbing the orientation of the polymer. Punching or shearing introduces local deformations and residual stresses near the cut edges. These may cause shape aberrations in the products as well as adversely affect further processing.

This project mainly focuses on the microstructures characterization of the polymer washers by using melts and cooling process to produce the samples. Polytetrafluoroethylene (PTFE), Polypropylene (PP) and Polyethylene (PE) is the examples of the polymer washer which can reduce friction, wear and energy consumption of machinery.

1.3 Objective

1. To study the microstructure characteristics of the plate polymer washer by using several laboratory experiments.

2. To compare the advantages and disadvantages of the polymer washer produced by the novel and existing punching techniques.

1.4 Scope of Study

The whole project would begin with the knowledge gathering and theoretical studies. Analyzing the existing method would be the fundamental step in this project. The step will be followed by investigate the limitation of the method and the effect on the polymer washer microstructure. By developing the new technique to produce the polymer washer, several lab experiments will be carried out to determine the effectiveness of the method. The experiments include the testing on the hardness, mechanical strength and polymer microstructure. The methodology will be developed according to the step-by-step procedures from identifying, extracting, and analyzing the microstructure characteristics of the plate polymer washer. Meanwhile, further research and development would be continuously practiced to ensure satisfactory results are achieved. The variables to produce the polymer washer are as shown as in Table 1 below.

Table 1: Parameter of the sample of polymer washer

External diameter of polymer washer, (OD)	(12 – 15) mm
Internal diameter, (ID)	(5 – 6) mm
Thickness of polymer washer, t	(1 – 2) mm
Temperature of solder, T	(400 – 450) °C
Speed of penetration, v	(4 – 5) s
Washer material	Laboratory experiment

CHAPTER 2

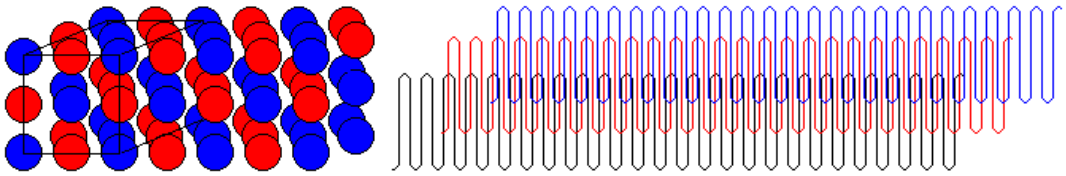
LITERATURE REVIEW

2.1 Polymer structure (morphology) [4,19]

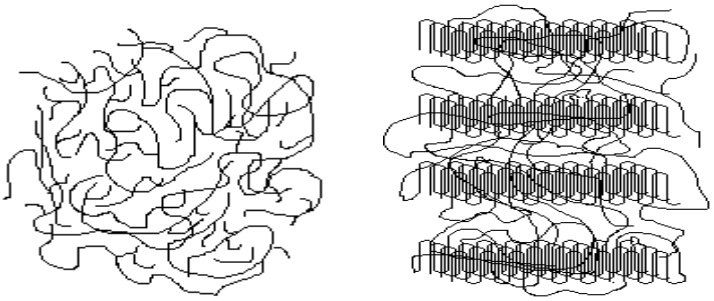
2.1.1 Amorphous or Crystalline [4,19]

Structurally, polymers in solid state may be amorphous or crystalline as shown in Figure 6. When polymers are cooled from molten state or concentrated from the solution, molecules are often attracted to each other and tend to aggregate as closely as possible into a solid with the least possible potential energy. For some polymers, in the process of forming a solid, individual chains are folded and packed regularly in an orderly fashion. The resulting solid is a crystalline polymer with a long range, three dimensional, ordered arrangements. However, since the polymer chains are very long, it is impossible for the chains to fit into a perfect arrangement equivalent to that observed in low molecular weight materials. A measure of imperfection always exists. The degree of crystalline is the fraction of the total polymer in the crystalline regions, may vary from a few percentages to about 90% depending on the crystallization conditions. Examples of crystalline polymer include polyethylene (PE), polyacrylonitrile and polytetrafluoroethylene (PTFE). In contrast to crystalline polymer, amorphous polymers possess chains that are incapable of ordered arrangement. They are characterized in the solid state by a short range order of repeating units. These

polymers vitrify, forming an amorphous glassy solid in which the molecular chains are arranged at random and even entangled.



(a)



(b) (c)

Figure 6: Polymer structure (a) crystalline (b) amorphous (c) semicrystalline structure[4]

2.1.2 Factors that affect the polymer microstructure [5,16]

A. Morphology of polymers crystallized from the melt (Heating process) [5,16]

Jemyung Park, Kyuyoung Eom, Ohjun Kwon, and Sangsun Woo had reported the morphology of polymers crystallized from the melt. This paper reveal when isostatic polypropylene (iPP) polymer is melted then cooled, it can recrystallize with process variable such as temperature, rate of cooling, pressure and addictive affected the nature of structure formed. Two types of microstructure observed for semicrystallization of bulk polymer are spherulites and row nucleated textures. Bulk of crystallized is consists of microscopic unit called spherulites which are formed during crystallization under quiescent condition. Spherulites are composed of lamellae structure which each lamella

is seen as a flat ribbon. Lamellae are the fundamental building units for the formation of crystalline micro structures.

One axis of each lamella extends forming a lamellar fibril. These lamellar fibrils now grow radially from a central nucleus but it has a tendency to twist, diverge and branch during growth. Lamellae usually have dimensions of 1 micro in length and 100A in thickness. [5]

This paper also stated that the spherulites exhibit radially symmetric growth of the lamellae from a centre nucleus with the molecular chain direction perpendicular to growth direction. The crystalline or laminar thickness is depending on the molecular weight of the polymer. The size and number of the spherulites is controlled by nucleation. Sphereulites are smaller and more numerous if they more growth nuclei or larger if slow cooled or isothermally crystallize. [5]

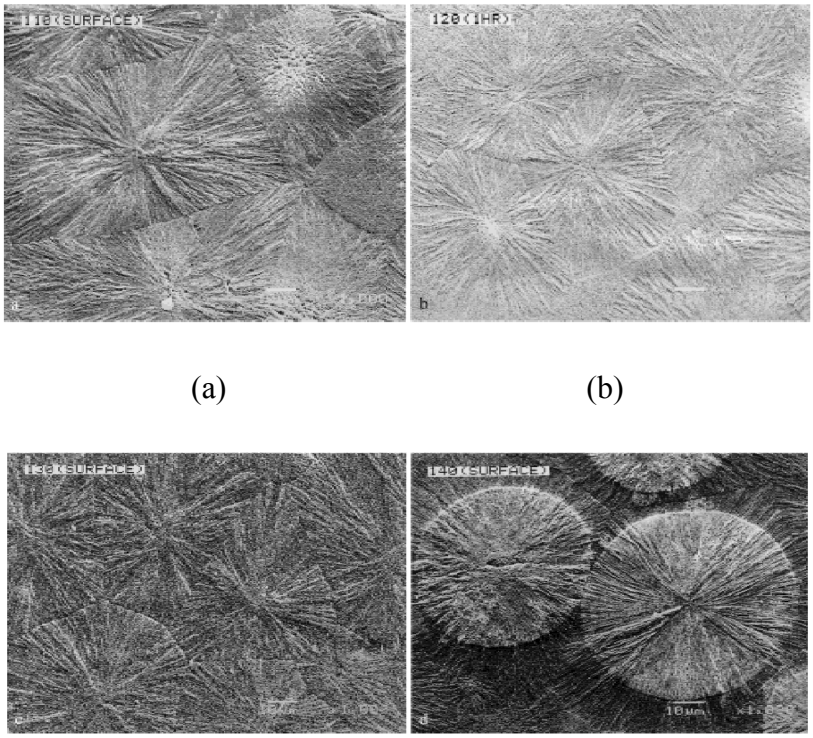
i. Spherulitic Morphologies of Various Crystallazation Temperature. [5]

Jemyung Park, Kyuyoung Eom, Ohjun Kwon, and Sangsun Woo reported that it is possible to predict the mechanism of spherulite growth in the investigation of spherulites morphology from different crystallization temperature (100°C ~ 140°C).

From Figure 7, the isothermal crystallized isostatic polypropylene (iPP) sample at 130°C had a high crystalline order which shows that the crystallization time is enough to grow each spherulites at 130°C. However, more time needed of crystallization temperature at 140°C because each spherulitic are broader than other and size of spherulites was relatively small.

This paper reveal that in the initial stage of crystallization, the spherulites were grown in a radial direction without any changes in the spherical morphology; the polygonal morphology was revealed due to the collision with the nearest spherulites during growth. The spherulite morphologies, crystallized at 110 ~ 130°C, were ypical a-spherulites according to their

growth pattern and dark image. The spherulite size was decreased as the crystallization temperature was increased. [5]



(a) (b)

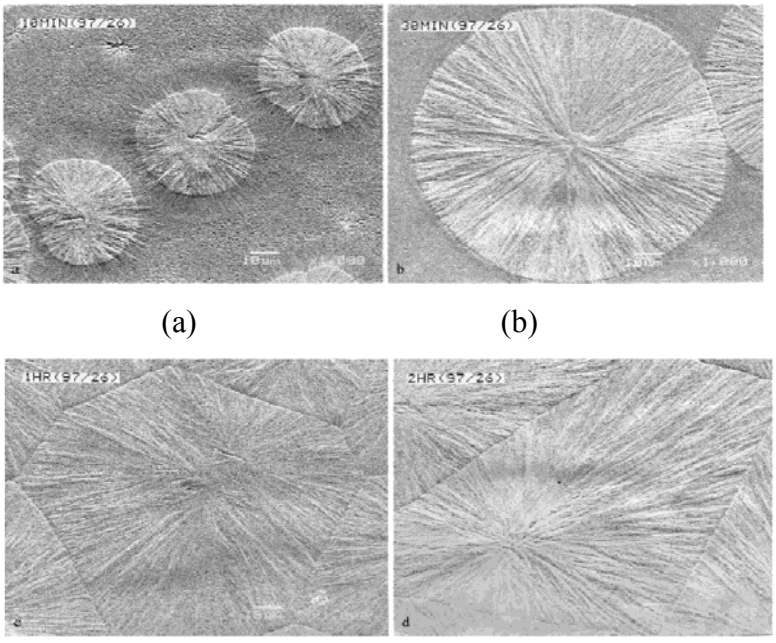
(c) (d)

Figure 7: Spherulite morphology of iPP crystallized at different crystallization temperatures (1 hr). (a): 110°C; (b): 120°C; (c): 130°C, (d): 140°C. [5]

ii. Spherulitic Morphologies of Various Crystallzation Times [5]

Jemyung Park, Kyuyoung Eom, Ohjun Kwon, and Sangsun also had reported the spherulitic morphologies of various crystallzation times which the isothermal temperature was maintained at 130C. Figure 8 show the resulting morphologies and crystal structure of each crystallization times. The crystallized sample for 10 minutes showed the initial stage of spherulites growth as shown in Figure 8(a). As the crystallization time went to 30 min, the size of iPP spherulites were enlarged from 20 ~ 30 μm to 100 μm. Most of samples had radial growth pattern and dendrite

morphology with a dark image. The bright region is the crystalline part of isostatic polypropylene (iPP) and the dark area represents the amorphous part. From Figure 8, it shows the radial growth of lamellae and each grown lamellar have a crosshatched component or daughter lamellae which correspond with tangential growth of lamellar.



(a) (b)

(c) (d)

Figure 8: Spherulite morphology of iPP at different crystallization times (130C): (a) 10 min (b) 30 min (c) 60 min (d) 120 min. [5]

B. Infuenced of Melt-shearing process on polypropylene microstructure [8]

Ellen and Arianna (2002) had reported that the processing of semi-crystalline polymers such as polyethylene (PE) or polypropylene (PP) involves the shaping of the molten material in moulds and dies by injection moulding methods or film blowing and fibre spinning techniques. Once it is processed, the polymer shape is stabilised via crystallization, controlling and determining the aesthetic and mechanical properties of the final material. However, a procedure like injection moulding and fibre spinning impose

shear flow into the polymer, this, in turn affects the crystallization kinetics and thus morphology. The rate of crystallization is profoundly altered by the influence of flow due to the alignment or orientation of segments of the molecular chains. This orientation of the chains is believed to affect the crystallization kinetics by providing sites where nucleation is promoted (in the flow direction). From these nucleation sites oriented lamellae grow radially, perpendicular to the flow direction. The development of oriented lamellar morphologies in bulk polymers has been termed the 'shish kebab' model, where the shish are the portions of oriented molecules and the kebabs are the outgrowing lamellae.

Figure 9 shows the development of row-nuclei when there is a melt-shear process undergo by the polymer sample. Melt-shear process caused development of row-nuclei in the form of microfibrillar bundles and promoted the epitaxial growth of folded chain lamellae that filled the space normal (perpendicular) to the row-nuclei, resulting in a supramolecular structure of cylindrical symmetry or cylindrites. In contrast, quiescent melt crystallization shows only spherulitic structures with folded chain lamellae. The rate of crystallization is profoundly altered by the influence of flow due to the alignment or orientation of molecular chains. [8]

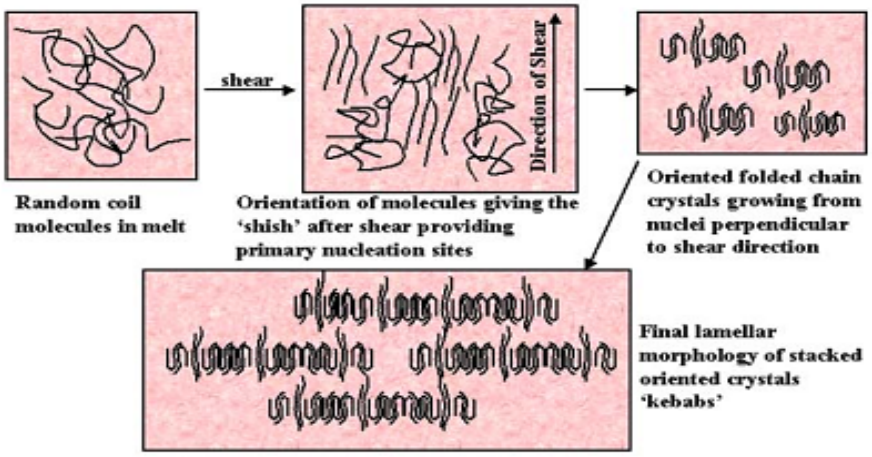


Figure 9: 'Shish kebab' development during shear-induced crystallization [8]

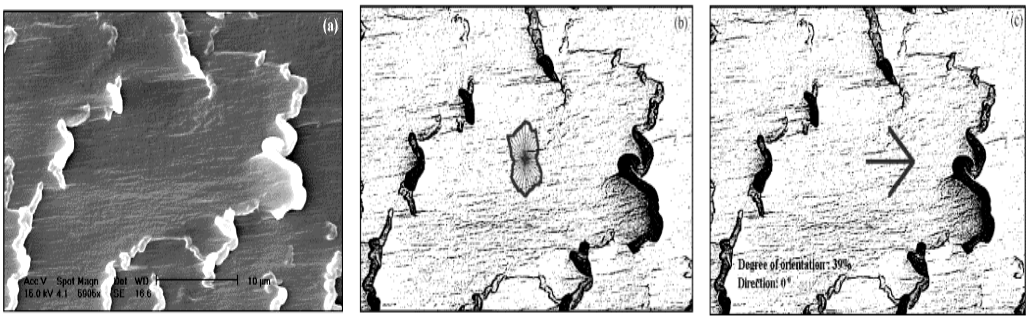
This orientation of chains will affect the crystallization kinetics by providing sites where nucleation is promotes in the flow direction. From the nucleation sites oriented lamellae grow radially perpendicular to the shear flow direction. The developments of oriented morphologies in bulk polymers have been termed the 'shish kebab' model which the shish are the portions of oriented molecules and kebabs are the outgrowing lamellae as shown in Figure 9. [8]

C. Isostatic polypropylene (i-PP) lamellar fibril behaviors under deformation (compression process). [9]

Giovanna, Maria and Dimitrios had reported the microstructural orientation of isostatic polypropylene (i-PP) studied by computerized scanning electron microscopy (SEM) image analysis. In this case study, the plane strain compression in channel die as in Figure 10 was chosen as deformation mode. The approach is suitable for deformation because it is homogeneous and avoids spurious cavitations phenomena such as micronecking observed during uniaxial drawing. Samples from core region of injection molding isostatic polypropylene (i-PP) (Mw = 438 200 g/mol; Mn = 90 000; Mw/Mn = 5.9) with 3 mm thickness, 4.7 mm width and 4.7 mm length were plastically deformed at ambient temperature using compression pressures of 1349 MPa and 2699 MPa. [9]

The samples were placed in a deep channel die and then, they were deformed. The upper and bottom surfaces of the chamber were polished with a very fine alumina powder (1.0 μm), avoiding any impurities during deformation. The samples were placed in liquid nitrogen for 10 minute and then rapidly fractured. Afterwards, the specimen were fixed on a stub and metalized with a thin gold layer using sputter coater in order to eliminate any desirable effect during SEM observations. Figure 10(a) below shows SEM image from the isostatic polypropylene (i-PP) sample after deformation at 1349 MPa and Figure 10(b) is the binary image with the rose of intercepts from the corresponding structure orientation. Binary image formed only black and white images. The images were used to determine orientation through the rose of the number intercepts and the

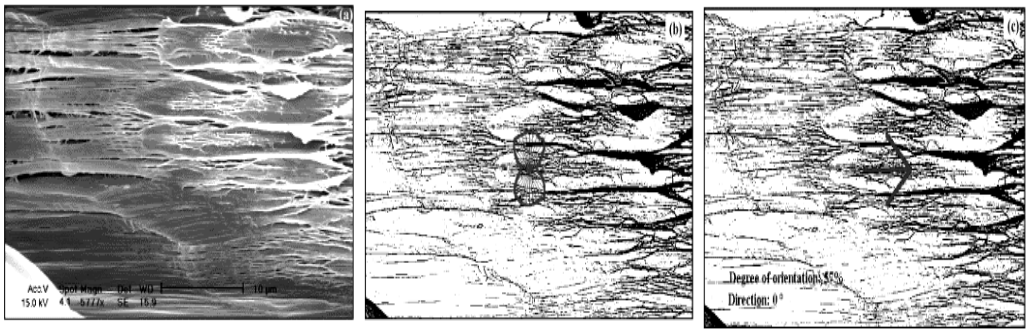
percent of orientation. In Figure 10(c), it shows a fibrillar structure observed with a degree of orientation about 39%. [9]



(a) (b) (c)

Figure 10: SEM image of i-PP microstructure under deformation pressure 1349 MPa. (a) SEM image after deformation (b) binary image (c) fibrillar structure. [9]

The effect is still more remarkable after compression at 2699 MPa, which is the formations of two petals are clearly and well defined. The evaluation of Figure 11(a) (SEM image), Figure 11(b) (processed image) and Figure 11(c) show a very significant degree of microlamellar orientation of 57%. [9]



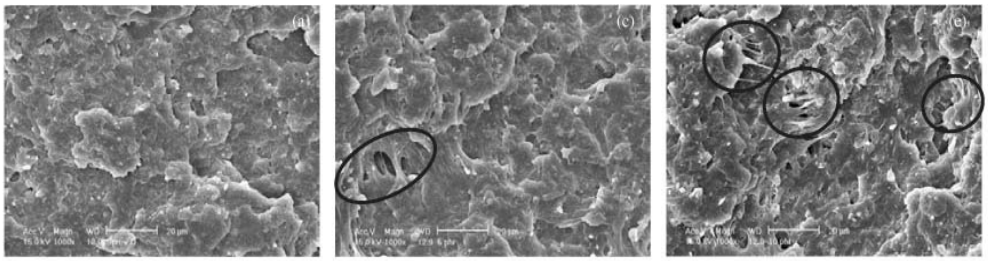
(a) (b) (c)

Figure 11: SEM image of i-PP microstructure under strong compression pressure. (a) microfibrillar under SEM image. (b) Processed image (c) significant degree of microfibrillar orientation of 57%. [9]

As a result, the image processing in the i-PP was easy to verify that increasing of the applied deformation pressure, causes a significant increase in the microstructure degree of the orientation. The microstructure changes from isotropic structure of the undeformed samples; Figure 10 shows lamellar at a moderate deformation (1349 MPa UCR = 65%) and to fibrillar (Figure 11) for the strong deformations (2699 MPa UCR = 90%). [9]

D. Microstructure of thermoplastic elastomer blends from waste tire powder and two polyolefins, Viz. maleic anhydride grafted polypropylene (PP-g-MA) and Low Density Polyethylene (LDPE). [10]

Jin Kuk Kim, Sung Hyo Lee and Maridass had reported a comparative study of effect of compatibilization agent on waste ground rubber tire and polyolefin blends. The thermoplastic polymer blends was developed from waste tire powder, (PP-g-MA) and (LDPE). The blends were processed using a twin-screw extruder equipped with a well-designed screw configuration. Significant improvement in the mechanical properties was obtained with the addition of maleic anhydride-grafted styrene-ethylene-butylene-styrene (SEBS-g-MA) as a compatibilizer in waste tire powder / polyolefin blends. In this case study, the effect of SEBS-g-MA compatibilizer on LDPE / waste tire powder and PP-g-MA/waste tire powder blends was studied by using the scanning electron microscopy (SEM) as shown in Figure 12. [10]



(a) (b) (c)

Figure 12: SEM micrographs of PP-g-MA blended with waste rubber and their blends with different amount of SEBS-g-MA content. (a) waste rubber : PP-g-MA (65:35); (b) 65:35, 5% SEBS-g-MA; (c) 65:35, 10% SEBS-g-MA. [10]

Figure 12 and Figure 13 shows the SEM micrographs of different olefin/ waste rubber and LDPE/ waste rubber blended with different amounts of SEBS-g-MA. The blends containing SEBS-g-MA showed better morphology as can be seen in Figure 12 (b), (c) and Figure 13 (b) and (c).

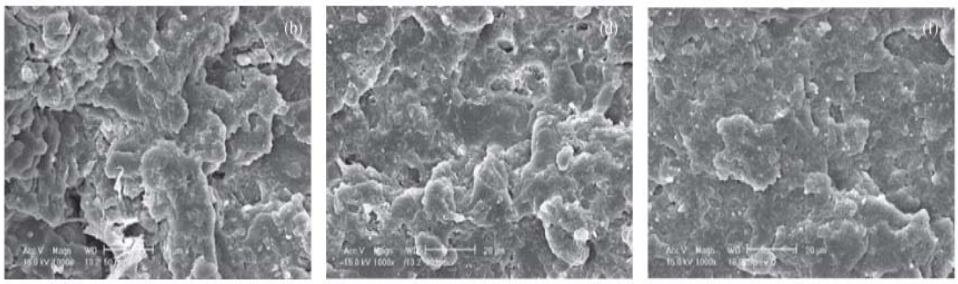


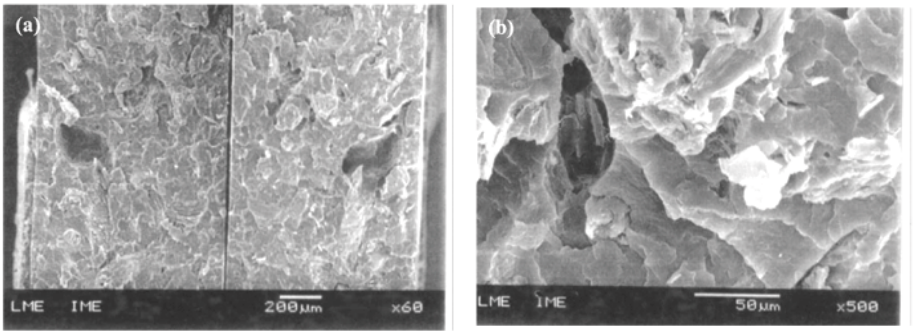
Figure 13: SEM micrographs of LDPE blended with waste rubber and their blends with different amount of SEBS-g-MA content. (a) waste rubber : LDPE (65:35); (b) 65:35, 5% SEBS-g-MA; (c) 65:35, 10% SEBS-g-MA. The bar corresponds to 20μm.[10]

As can be seen in all blends, rubber particles constitute the dispersed phase with a continuous thermoplastic matrix. With the addition of compatibilizer, the surface properties of the blends were further improved. [10]

E. Analysis of the fracture behavior of polypropylene – sawdust composites. [11,17]

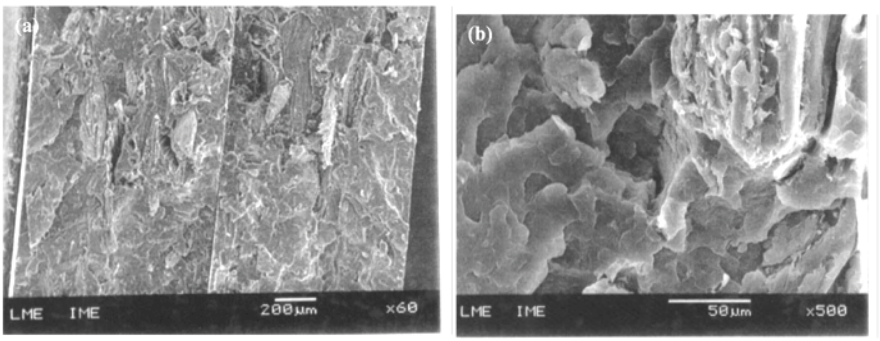
Joao C, Fernanda M. B. and Coutinhohais had reported the analysis of the fracture behavior of polypropylene blends with sawdust composites. In this case study, the composites of polypropylene (PP) plus maleated polypropylene (MAPP) filled with sawdust were prepared under fixed processing conditions (mixing temperature, mixing time and rate of rotation). The composites were fractured by tension which it performed at room temperature in a model 4204 Instron Universal Testing Machine with a load cell of 1 kN, at a crosshead speed of 5 mm/min using rectangular specimen of 100 mm x 8 mm, punched out from the molded plates. As shows in Figure 14, the failure is due to fiber pull-out accompanied by tearing of the matrix; the pull-out increases with the MAPP content.

Figures 14 to 16 present SEM photomicrographs of the tensile fracture surfaces of the PP/MAPP/sawdust composites. The MAPP addition to PP-sawdust composite does not modify the basic fracture mechanism of the PP matrix. The composite failed, predominantly, by transversal fracture in the plane of the matrix, showing similar fracture features: surface roughness and the presence of conic marks. However, one can see that the increase in the MAPP content and the addition of sawdust produce modifications in the matrix surface with pull-out and breakage of the fibers. In this manner the fracture morphology of the composites shows some differences according the PP/MAPP ratios. The SEM photomicrographs of the 98/02 composites (Figure 14(a) and 14(b)) show an effective fiber-matrix adhesion. It can be seen, that a layer of the matrix material that have been pulled out together with the fiber. This is an indication that at this MAPP content occurs an effective interaction between the sawdust filler and the PP/MAPP matrix.



(a) (b)

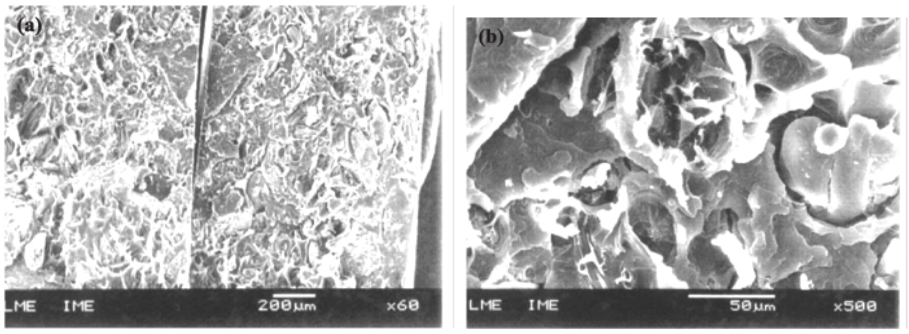
Figure 14: SEM photographs of tensile fracture surfaces of 95/02 PP/MAPP composite: (a) image view of signification of 60x (b) signification of 500x. [11]



(a) (b)

Figure 15: SEM photographs of tensile fracture surfaces of 95/05 PP/MAPP composite: (a) image view of signification of 60x (b) signification of 500x. [11]

In Figure 15, 95/05 composites it is observed the existence of cracks in the matrix-filler interface and, in consequence, a worse interfacial adhesion between the sawdust and the PP/MAPP matrix. In addition to fiber pull-out, as the mode of fracture, fiber breakage can also be seen in the samples.



(a) (b)

Figure 16: SEM photographs of tensile fracture surfaces of 95/10 PP/MAPP composite: (a) image view of signification of 60x (b) signification of 500x. [11]

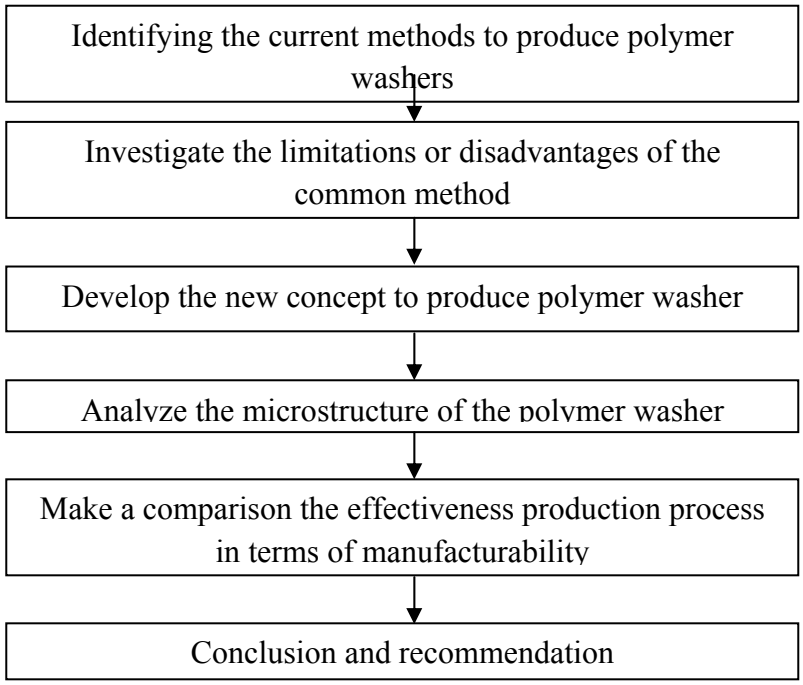
In Figure 16, the photomicrographs of the 90/10 composites exhibit cracks and a concentration of holes left after the fibers are pulled out from the matrix. The holes

proximity indicates that the sawdust exist in the form of fiber bundles that did not provide an efficient stress transfer from the matrix to the fiber.

CHAPTER 3

METHODOLOGY

In order to ensure the project complete smoothly, a flow chart of methodology have been develop by following the Gantt chart schedule as in Appendixes. The methodology of the project shows from the initial steps of the project until it completed as shows in Flow chart 1 below.



Flow chart 1: The methodology of developing new washer production concept.

In the Flow chart 1 as above, the project is started with gathering the knowledge of the polymer formation, polymer washer process and polymer characteristics. The existing method to produce the polymer washer is analyzed by focusing on the disadvantages and limitation of the process on the polymer microstructure. Then, the new method is developed which is much easier and cheaper to investigate the performance and criteria of the polymer washer. This method uses a melting process and consequently enhances the strength of polymer when the polymer is heated. Finally, the washer produce by existing and the new methods are compared to each other and some recommendation has to be made due to the results of microstructure characterization of the polymer.

3.1 Process methodology

3.1.1 Melt and shearing process to produce polymer washer

There is an alternative method to produce a washer rather than use punching or shearing process. Instead of using a punch, a steel solder with the high temperature is applied to the polymer plate to produce a washer as shown in Figure 17. Thus, there will be zero of the wasteful of the material. The solder is holds in vertical position and penetrate the polymer plate until a hole is produced. The solder should be removing from the polymer immediately to avoid the washer stick to the plate. Applying the heat or melting the polymer modifies the microstructure of the polymer washer.

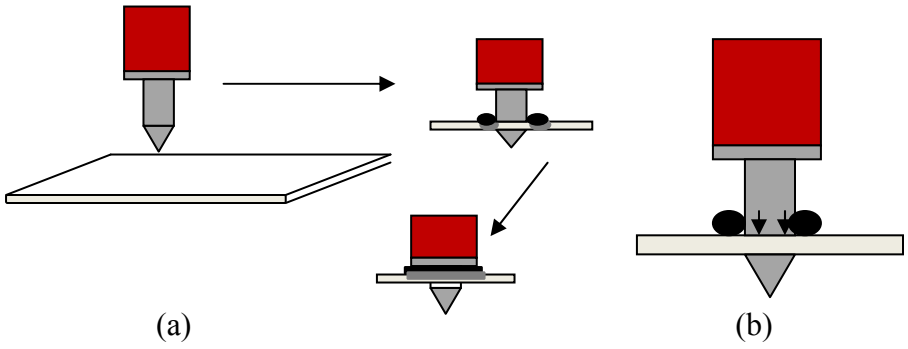


Figure 17: (a) The process of heating the polymer plate (b) Shearing on the polymer structure.

3.2 Sample Preparation [5]

The morphology of polymer has been widely investigated by electron microscope after the proper sample preparation techniques were developed. In this project, the permanganic etching technique of iPP has been used to prepare the sample for laboratory experiments. The etchant compositions are potassium permanganate, concentrated phosphoric acid and concentrated sulfuric acid (KMnO4/H3PO4/H2SO4). In order to prepare the sample preparation, several procedures have been conducted. [5]

1) Material safety data sheet (MSDS) for each chemical substances used have to read and understand first before preparing the acid permanganic. (PRECAUTION: Direct contact of potassium permanganate (KMnO4) with sulfuric acid and hydrogen peroxide may produce explosion (MSDS).)

2) Four 100ml beakers are needed to prepare the etchant solution and a cylinder to measure the required amount of the acid. (NOTE: In order to avoid any dangerous conditions, all of the etching procedures were performed at room temperature.)

3) To prepare the solution, 32.2 ml of sulphuric acid (H2SO4) is poured in the beakers.(NOTE: Never add water to this acid)

4) 16.2 ml of phosphoric acid is mixed with the sulphuric acid in the beakers. (NOTE: Never add water to this acid)

5) After the acid mixture has been produced, 1.5g of potassium permanganate is put in the mixture for each beaker.

6) Then, two polymer samples are placed in the etchant solution and leave it for 5 hours. (NOTE: Ensure the open part of the beaker is closed using parafilm.)

7) Step 3 to 6 is repeated for different time duration for the samples which are 7 hr, 20hr and 25 hrs.

8) Finally, the samples were taken out from the solution and carefully washed with distilled water, hydrogen peroxide, and acetone sonication in order to avoid any artifacts.

3.3 Laboratory Testing

A variety of lab techniques are used to determine the type of the polymer, the microstructure of polymers and its effectiveness to the polymers property.

3.3.2 Scanning Electron Microscopy (SEM) [12]

The scanning electron microscope (SEM) as in Figure 18 is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. [12]

Figure18: SEM opened sample chamber. [12]

Nonconductive specimens tend to charge when scanned by the electron beam especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample either by low vacuum sputter coating or by high vacuum evaporation. Conductive materials in current use for specimen coating include gold,

gold/palladium alloy, platinum, osmium, iridium, tungsten, chromium and graphite. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation.

Two reasons for coating, even when there is enough specimen conductivity to prevent charging, are to increase signal and surface resolution, especially with samples of low atomic number (Z). The improvement in resolution arises because backscattering and secondary electron emission near the surface are enhanced and thus an image of the surface is formed. [12]

3.3.3 X-Ray Diffraction (XRD) [13]

A diffractometer can be used to make a diffraction pattern of any crystalline solid. With a diffraction pattern an investigator, it can identify an unknown mineral, or characterize the atomic-scale structure of an already identified mineral. Diffraction occurs as waves interact with a regular structure whose repeat distance is about the same as the wavelength. The phenomenon is common in the natural world, and occurs across a broad range of scales. For example, light can be diffracted by a grating having scribed lines spaced on the order of a few thousand angstroms, about the wavelength of light. It happens that X-rays have wavelengths on the order of a few angstroms, the same as typical interatomic distances in crystalline solids. That means X-rays can be diffracted from minerals which, by definition, are crystalline and have regularly repeating atomic structures. When certain geometric requirements are met, X-rays scattered from a crystalline solid can constructively interfere, producing a diffracted beam. [13]

3.3.4 Fourier transform infrared (FTIR) spectroscopy [14,18]

Infrared spectroscopy (IR spectroscopy) is the subset of spectroscopy that deals with the infrared region of the electromagnetic spectrum. It can be used to identify compounds or investigate sample composition. In order to conduct FTIR

testing, the polymer sample is required to have a thin film size. In order to prepare the thin film from the solid polymer washer, the washer is compress between two platens which both platens are heated to a certain temperature. [14]

CHAPTER 4

RESULTS AND DISCUSSION

In order to study the morphology of the polymer sample microstructure after deformation, several laboratory experiments have been conducted. The polymer washer samples are prepared with suitable sample preparation before it was tested under X-Ray diffraction (XRD) and Scanning electron microscopy (SEM). In order to determine the composition of the polymer sample, FTIR testing is first conducted.

I. Fourier transform infrared (FTIR) spectroscopy

The results of the FTIR testing for polymer sample gave the pattern as shown in Figure 19. In order to determine the composition of the polymer sample, the type of the polymer always use to produce the chopping board is investigate. Basically, many polymer compounds are a mixture of two or more materials.

From the findings, two most common the manufacturer use to produce the chopping board are mixture of polypropylene and polyethylene with some others blends composites. First, the results are compared with the FTIR graph for the polypropylene (PP) and polyethylene (PE) either the results are match for the polymer sample FTIR graph.

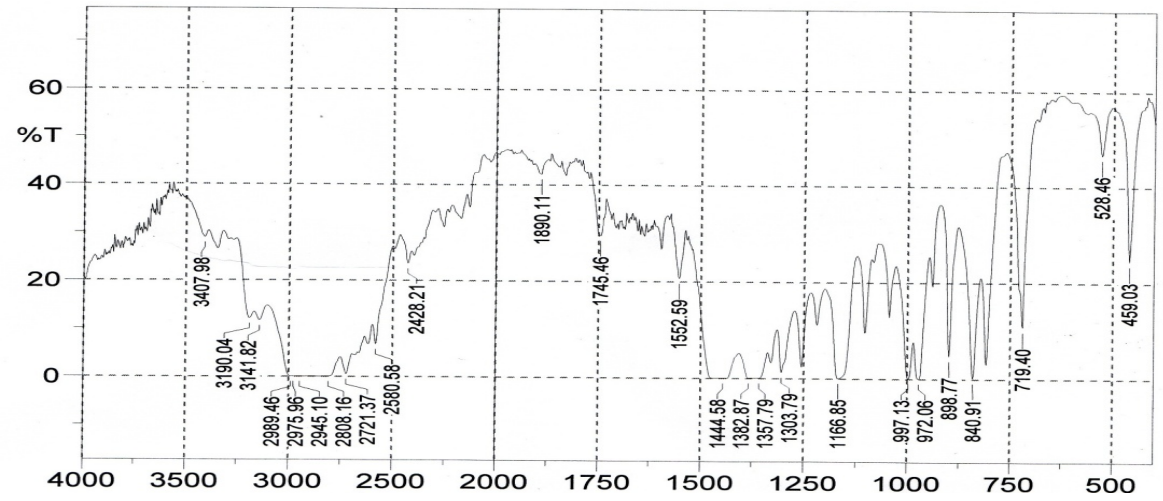
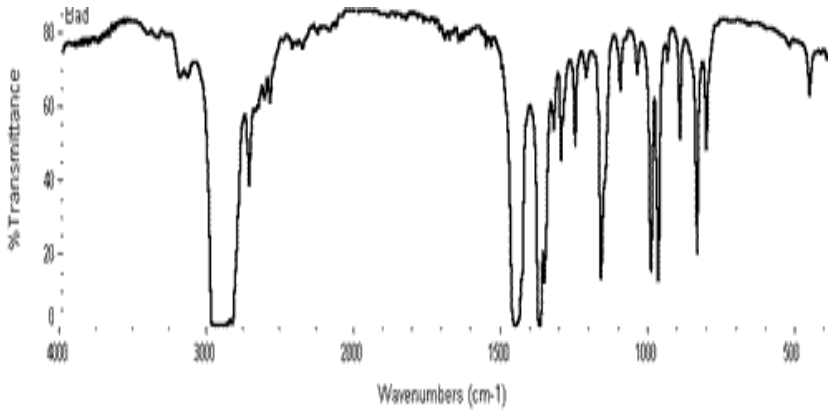
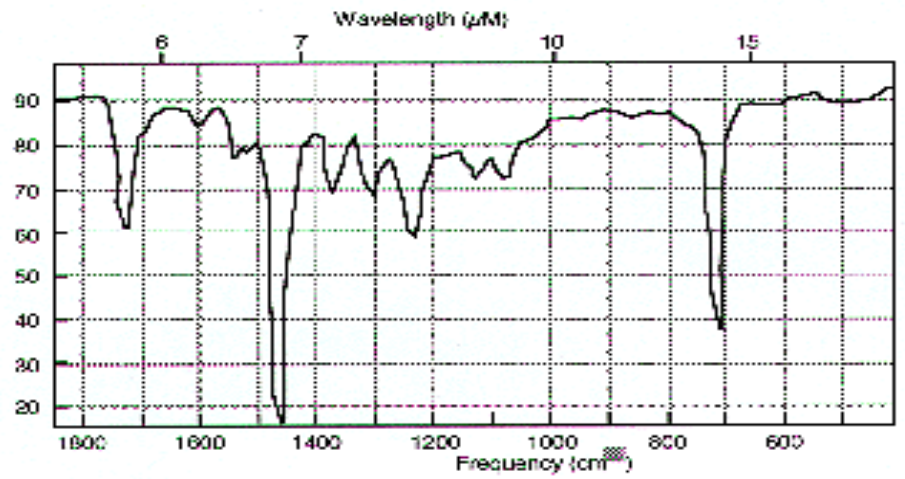


Figure 19: FTIR graph for polymer washer sample.

Refer the actual scale of Figure 19 in the ***Appendix 3***. From the observation, the peaks in an absorbance spectrum represent the wavelengths where the vibrating chemical bonds absorb infrared radiation. These correspond to particular types of bonds.



(a)



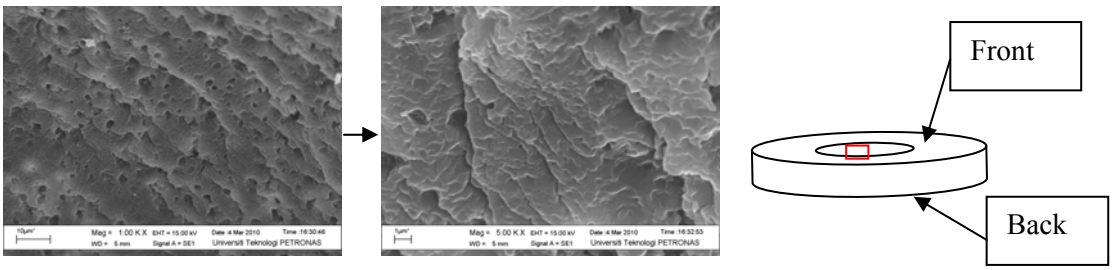
(b)

Figure 20: FTIR graph for (a) polypropylene sample (b) Polyethylene sample [14]

After a comparison has been made with Figure 20, most of the absorption peaks are accounted for by a polymer identified chemically as a polypropylene and polyethylene. However, there are several absorption bands that appear in the sample spectrum that are not contained in the library match. FTIR alone cannot determine if this represents a separate materials mixed with the primary material. For this type of detail, more sophisticated techniques are needed.

II. Comparison of scanning electron microscopy (SEM) photomicrographs of polymer washer produce by melt-shearing and punching technique.

Permanganate etching technique has been developed (Olley and Bassett, 1989) to allow direct observation of the spherulitic and lamellar morphology at the microstructure level for SEM and OM. But, OM is impractical to use in this study because it is not suitable for structural characterization of bulk samples or thick specimens of polymer washer. The permanganate acid solution preferential etches the amorphous of the polymer in the spherulites which the lamellar structure then clearly appeared. From the results of SEM, polymer washer sample C which shown the most clear appearance of lamellar and microfibril are observed. There are four important surfaces of the washer to investigate which are at edge, surface near hole, inside hole and back surfaces. The figures also can be referred in the ***Appendix 2***.

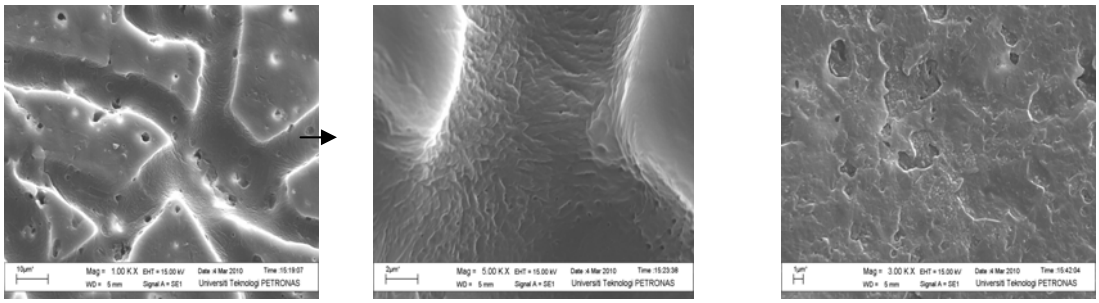


(a) (b)

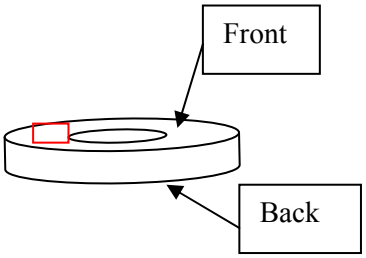
Figure 21: SEM image for inside hole of the polymer washer. (a) Magnification of image used is 1000x and 5000x (b) Schematic diagram of polymer washer.

Based on the Figure 21(a), it clearly shows the development of row nuclei in form of lamella fibril at the hole of the washer. The lamellar fibril is appeared due to the melt-shearing process which perform by the heating elements from the solder. Figure 21(a) have shown the distance from a stack of lamella to another lamella fibril is very close to each other which indicate that the hole is the high shear rate region. This molecular chain of orientation of lamella fibril will increase as the shear rate and temperature was increased. The toughness of polymer will be increasing as it exhibit high chain of orientation order or small gap between lamella fibril.

However, in Figure 22 (a) shows there is appearance of the amorphous region at the center surface of the polymer washer. This is because some part of the surfaces have not fully etches by the chemical etching agent which have been done during the sample preparation process. Moreover, there is a unwanted hole at the center surface of the polymer which may cause by the rusting surface of the bottom plate of solder. The unwanted hole will reduce the strength and stiffness of the polymer washer.



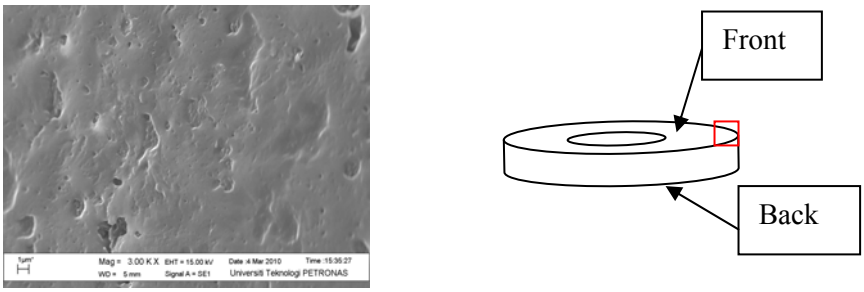
(a) (b)



(c)

Figure 22: SEM image at center surfaces of the samples of polymer washer. (a) Magnification of image used is 1000x and 5000x (b) different center surface area (c) Schematic diagram of polymer washer.

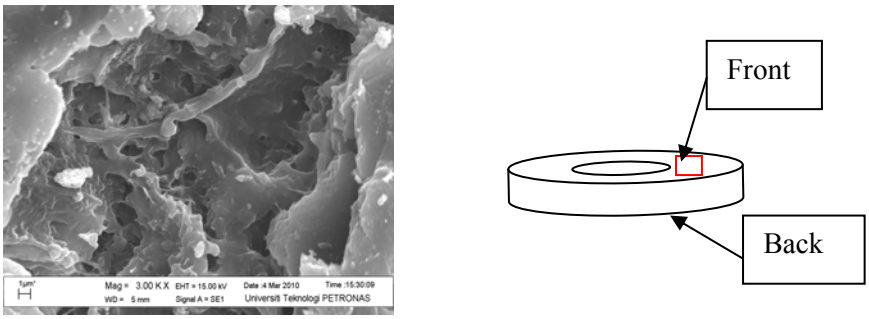
However, at magnification of 5000x for a deep surface of the polymer washer shows there is a development of the lamella structure but at very low molecular chain orientation. This is due to the low shear rate region for the center surface of the polymer washer. In Figure 22 (b), the image of the lamella fibril also cannot be really seen in this image because of low shear rate process.



(a) (b)

Figure 23: SEM image at edge surfaces of samples of polymer washer. (a) Magnification of image used is 3000x (b) Schematic diagram of polymer washer.

While for Figure 23 (a), it shows the microstructure for the edge surface of polymer washer. The development of lamellar fibril have a larger gap between adjacent lamellae or low chain orientation because it is a low shear rate region and tends to cool faster than region near the hole. The toughness of the polymer is lower at the edge surface compare to the inside hole surface.



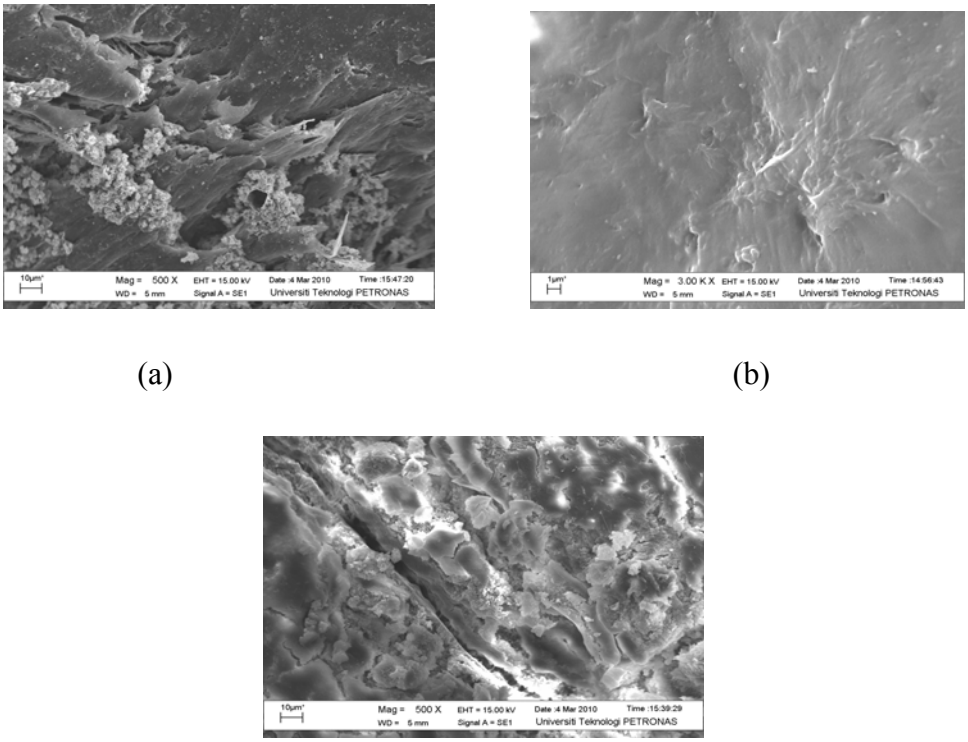
(a) (b)

Figure 24: SEM image at back surfaces of the samples of polymer washer.
(a) Magnification of image used is 3000x (b) Schematic diagram of polymer washer.

For Figure 24, the SEM image had shown the fracture surfaces at the back part of the polymer washer. This is because the bottom part of the washer undergo very high shear rate which caused by the solder and also at the unmelt surface. The friction between this

two surface lead to the breakage of the polymer structure and this will lowering the toughness of the polymer washer.

The results of the polymer washer produce by a novel technique shown earlier should be compared to the washer produce by a punching method. Punching process is conducted by producing the washer using drilling technique to get the hole and shape of washer. From the observations, there is a breakage of the polymer washer sample.



(a) (b)

(c)

Figure 25: The rupture image shown by polymer washer produce by punching method. (a) hole (b) center surface (c) near edge.

Figure 25 (a) shows that there is a rupture image of the lamella fibril structure at the region the hole. The failure of the lamella structure is due to the drilling process which the drill use a force to cut or tearing the polymer sample. For figure 25 (b), the stress or force from the drill cause the development of the microvoid at the center surface of the washer. The existence of the microvoid at the surface of polymer will affect the

performance of the washer to distribute the load for threaded fastener and to prevent the vibration of the tools. While for Figure 25 (c), there is fracture structure and a crack occur at the edge surface of the polymer. This crack may cause by the force of the cutter use in the drilling process. As a result, the toughness of the polymer washer will be lower and shorten the duration of washer life.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The polymer washer is observed to have better image of the microstructure when it undergo the melt-shearing process compared to punching technique. From the findings, the punching technique use to produce the polymer washer leads to breakage and cracks which this will decreasing the mechanical strength of the washer. Sample preparation need to be conducted before the microstructure of the polymer washer can be captured under Scanning Electron Microscopy (SEM). For bulk samples or thick polymer sample, sample preparation known as chemical etching technique has been used to etch the amorphous part of the polymer in spherulites. From the results, the polymer washers that undergo the melt-shearing process has developed a row-nuclei lamella fibril structure. The lamella fibril growth is perpendicular to the direction of the shear flow. Development of a row-nuclei lamella fibril is enhancing the mechanical strength of the washers.

In order to study the microstructure of the polymer washer, another experiment is conducted to determine the composition of the polymer. Fourier Transform Infrared Spectroscopy (FTIR) is used to identify the compounds of the washer sample. The results FTIR shown that the polymer washer is mixture of

polypropylene (PP) and polyethylene (PE) which possesses excellent properties such as lightweight, high tensile strength, impact resistant and high compressive strength.

5.2 RECOMMENDATION

The temperature and cooling rate are the factors that will affect the lamella fibril growth and size. The polymer washer produce by a melt-shearing process should be perform in several different solder temperature and cooling rate in order to achieve a better understanding of the microstructure image captured under SEM.

Hence, the best temperature can be determined to produce the good quality of the polymer washer.

CHAPTER 7

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APPENDIXES

GANTT CHART / MILESTONE

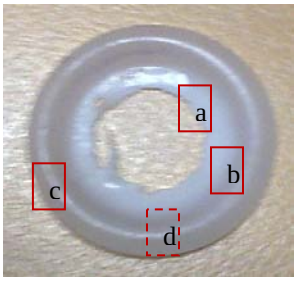
APPENDIX 1: Gantt Chart / Milestone of FYP 1

		Week No/Date													
No.	**Activities**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	**Selection of project topic**														
2	**Preliminary Research Project**														
3	**Submission of Preliminary Report**					**21/8**									
4	**Project Work**														
5	**Submission of Progress Report**								**8/9**						
6	**Seminar**								**11/9**						
7	**Project Work Continues**														
8	**Submission of Interim Report Final Draft**														**26/10**
9	**Oral Presentation**								2/11 – 13/11						

Appendix 2: Gantt Chart / Milestone of FYP II

No.	Activities	Week No/Date														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	**Project work** • Understanding the sample preparation technique • Prepared the permanganate acid solution • Test the sample under SEM								**Mid Sem**							
2	**Progress report 1**					**22/2**										
3	**Project work** • Understand the image occur from SEM • Investigate the sample microstructure type															
4	**Progress report 2**									**22/3**						
5	**Seminar**									**22/3 – 26/3**						
6	**Project work** • Compare the microstructure of the novel technique and punching process • Recommendation															
7	**Poster Submission**												**12/4**			
8	**Dissertation of final draft**															**3/5**
9	**Oral Presentation**									STUDY WEEK						
10	**Hardbound Dissertation**															**20/5**

Appendix 2: SCANNING ELECTRON MICROSCOPY (SEM)

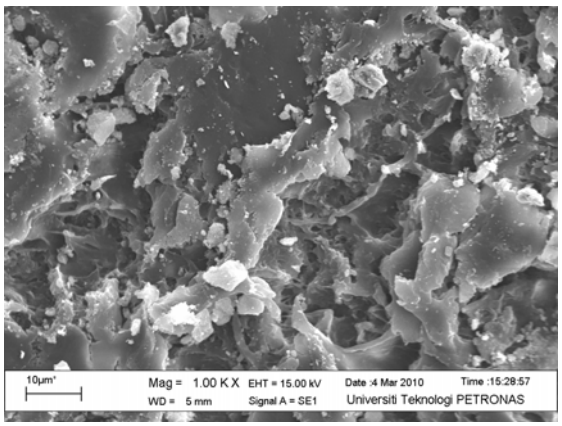


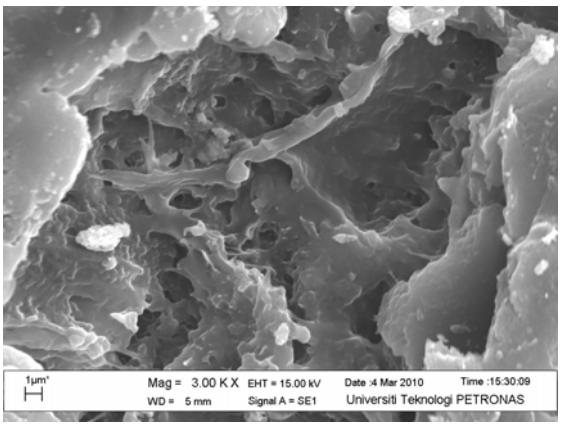
Polymer washer surface analyzed by SEM
a) hole
b) center
c) edge
d) back

<u>Polymer washer produced by A NOVEL TECHNIQUE (MELT-SHEARING PROCESS)</u>

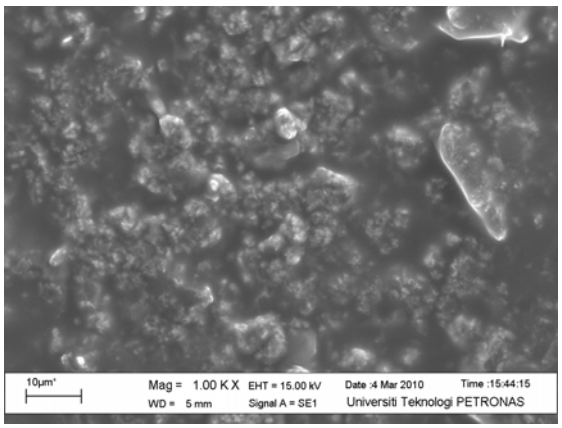
1) Polymer washer back surface: magnification of 50x, 1000x and 3000x

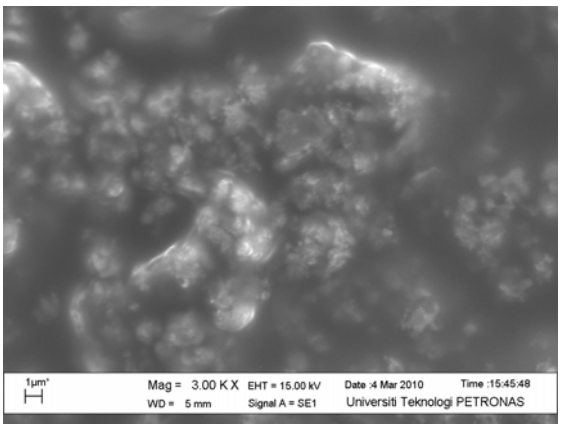
Sample A



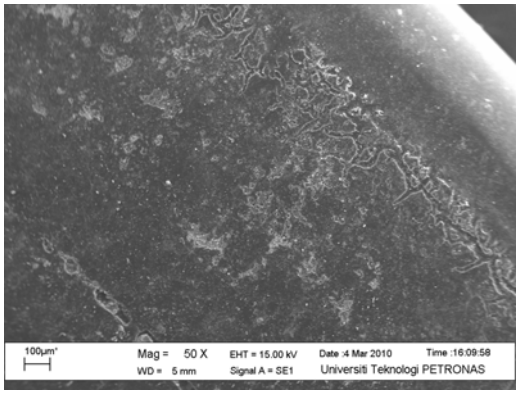


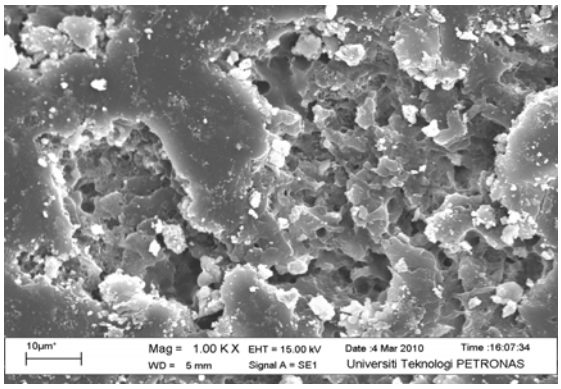
Sample B

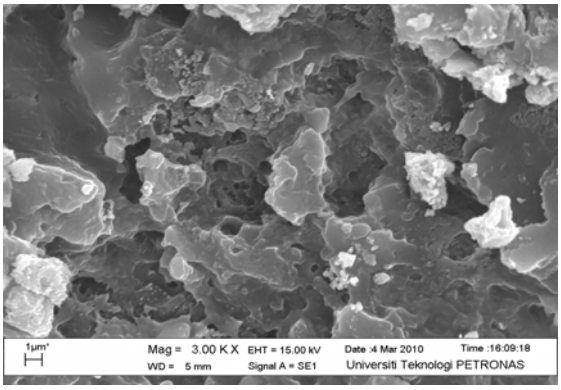




Sample C

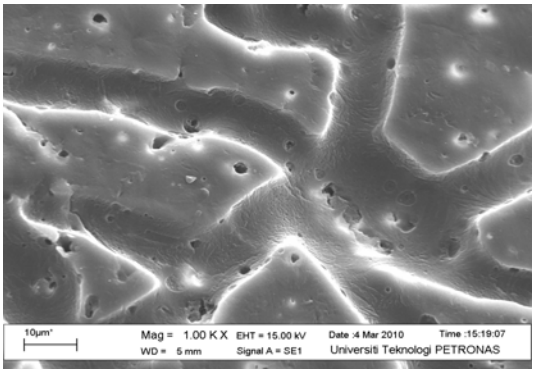


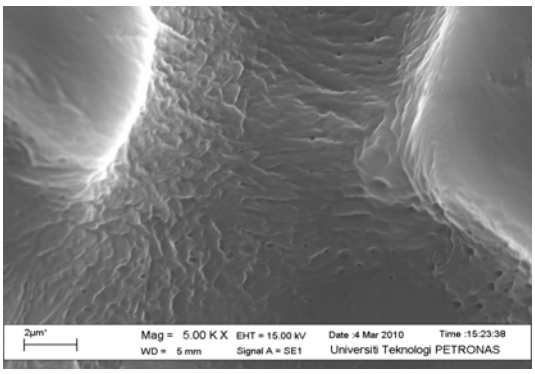




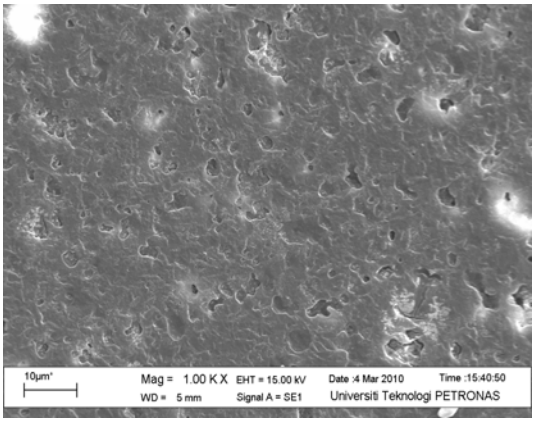
2) Polymer washer center surface: magnification of 1000x, 3000x and 5000x

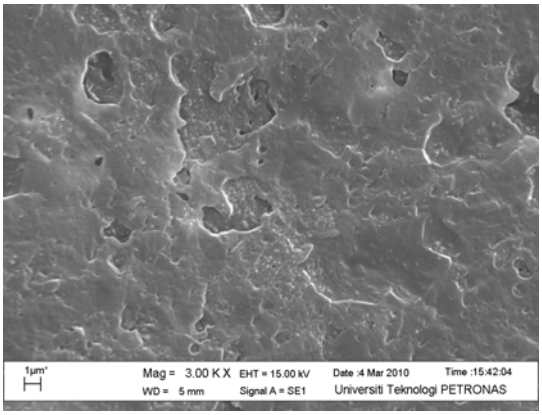
Sample A



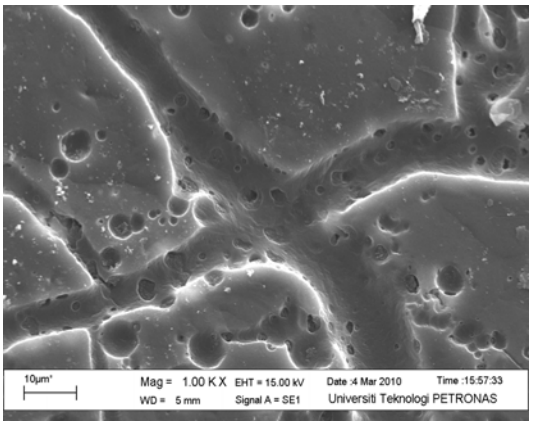


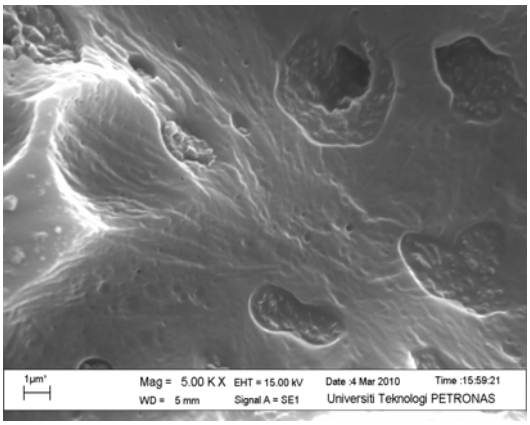
Sample B





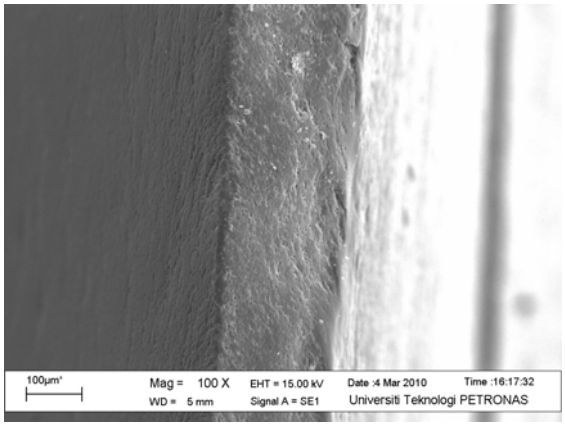
Sample C

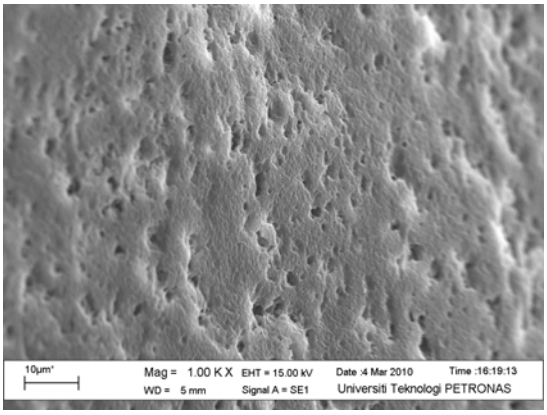


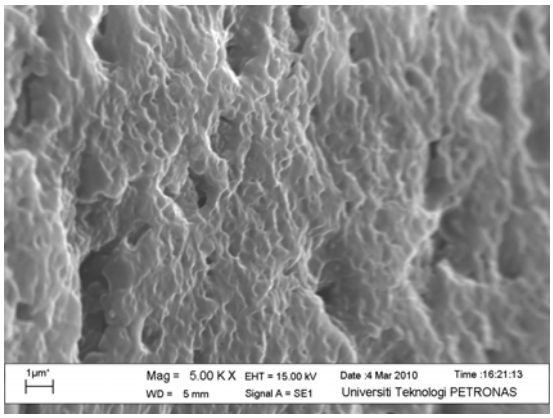


3) Polymer washer inside hole structure: magnification of 100x, 1000x and 5000x

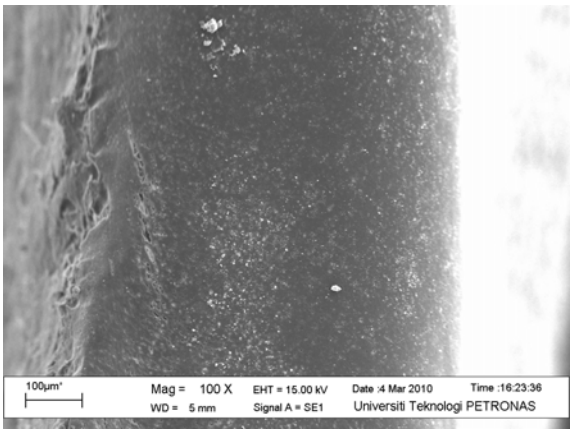
Sample A

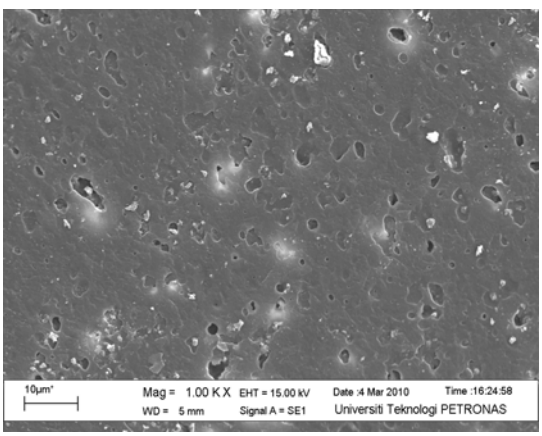


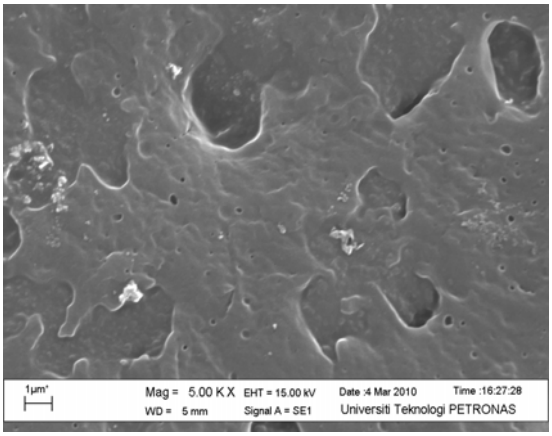




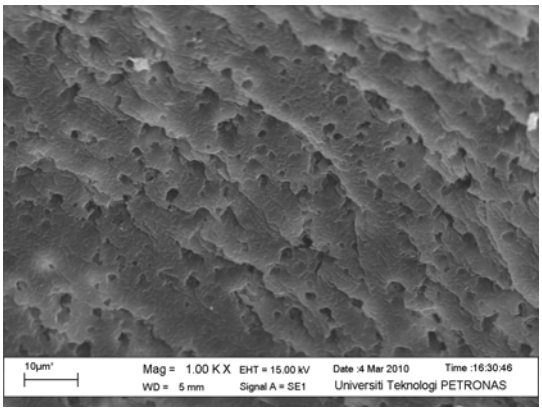
Sample B

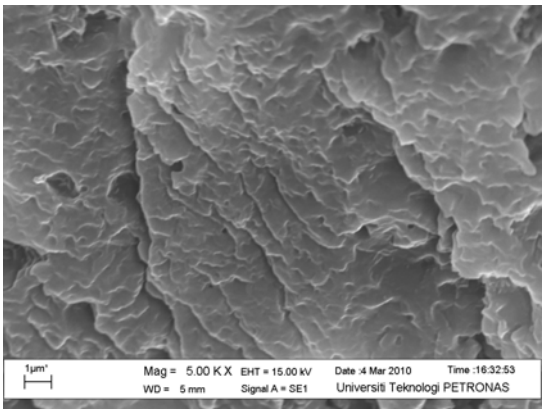






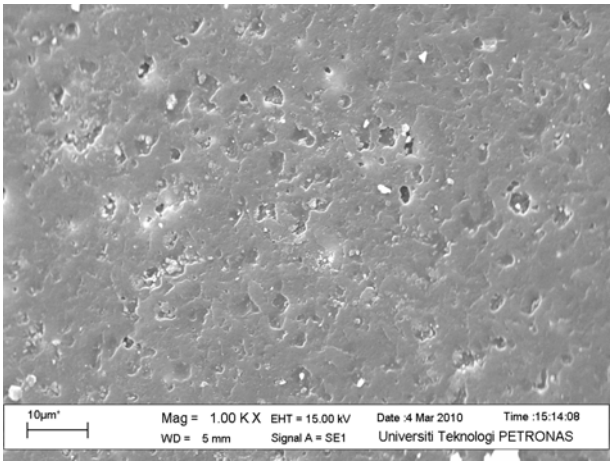
Sample C



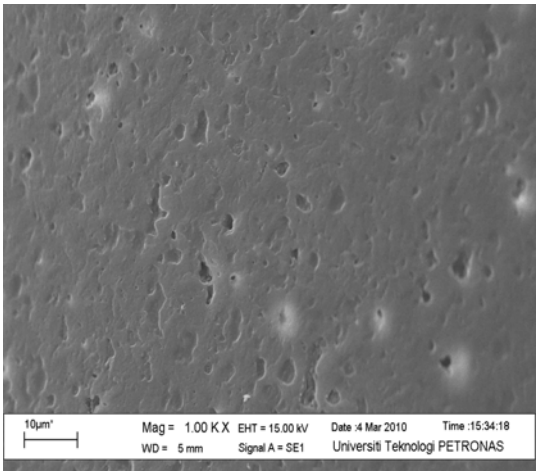


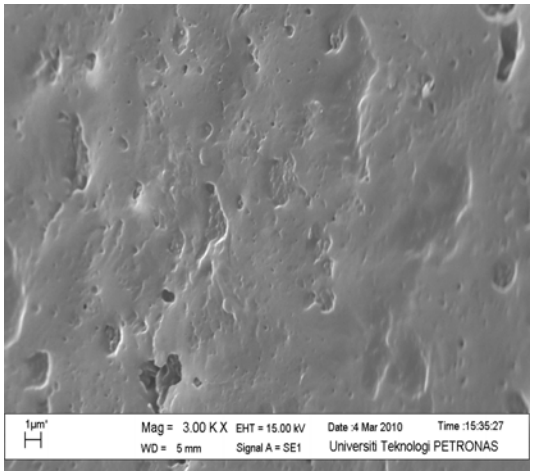
4) Polymer washer edge surface: magnification of 1000x and 3000x

Sample A

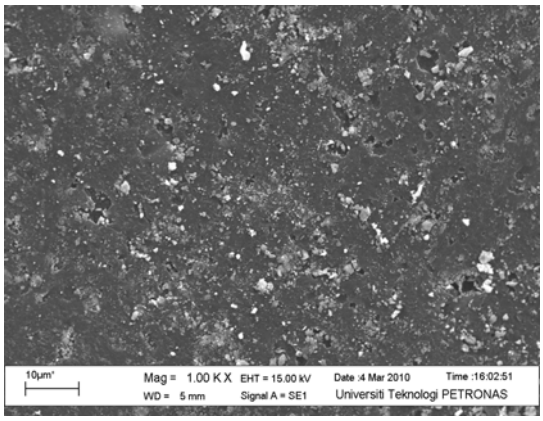


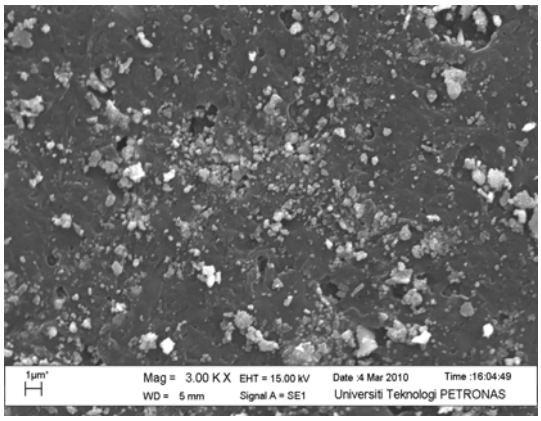
Sample B





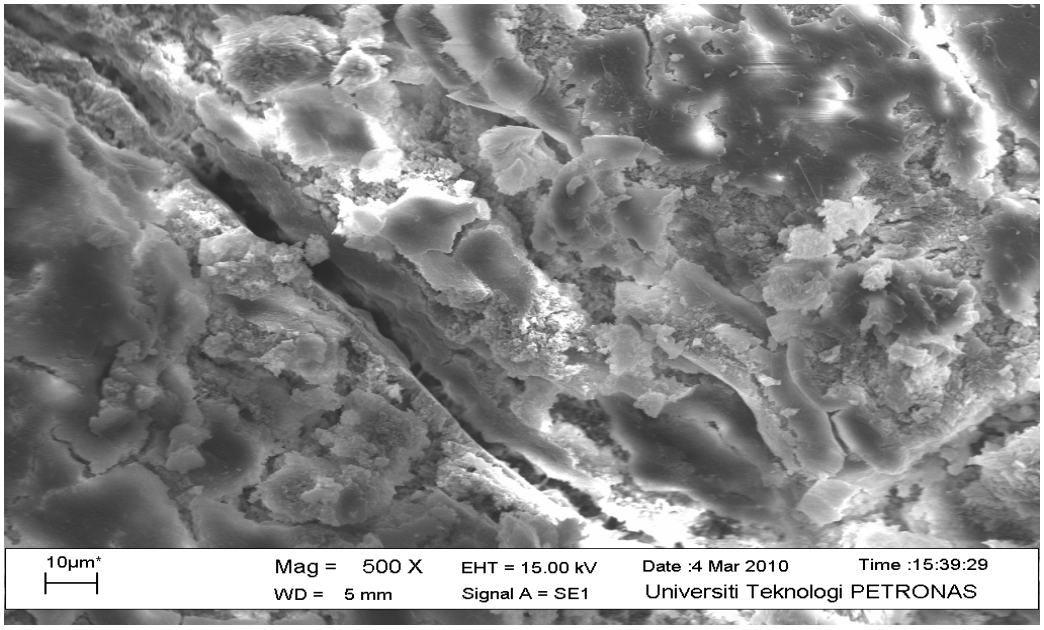
Sample C



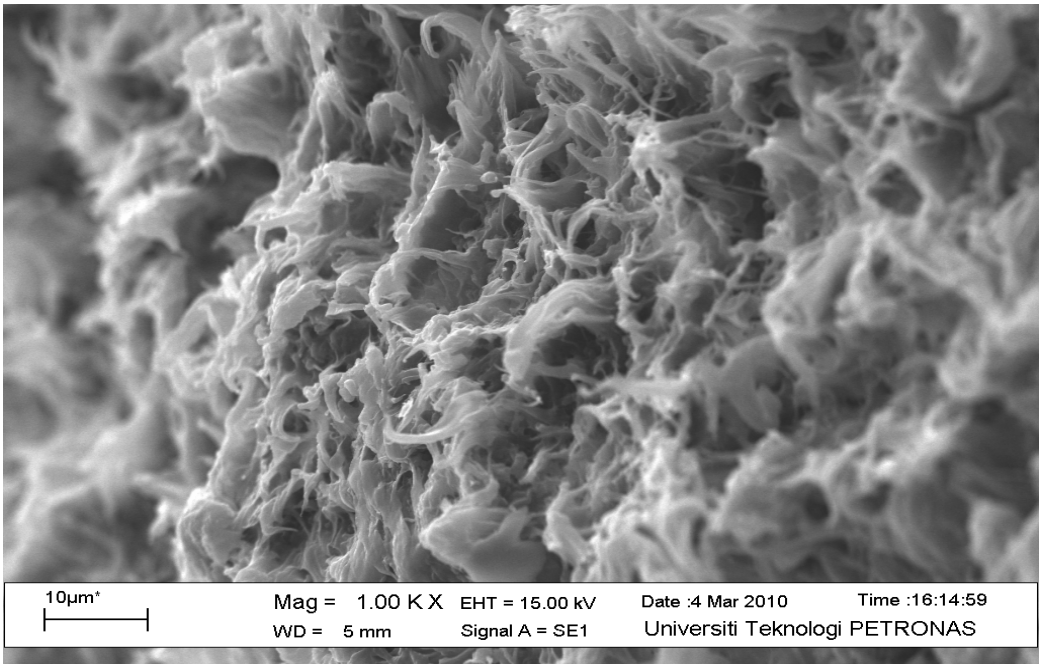


Polymer washer produced by PUNCHING METHOD

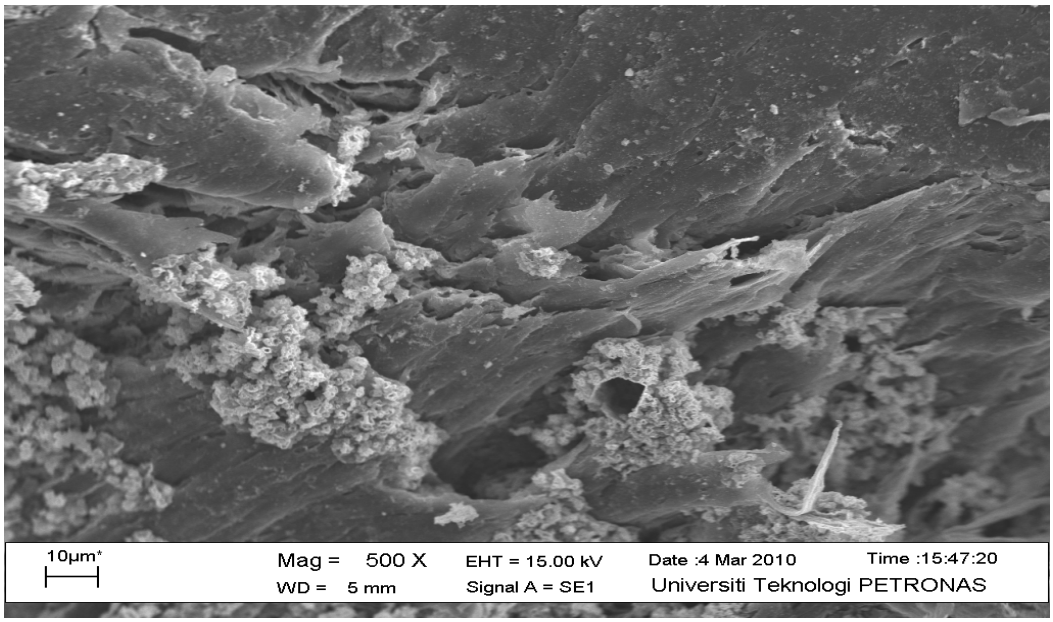
5) Polymer washer microstructure



Magnification of 500x for center washer front surface



Magnification of 1000x for hole region washer front surface



Magnification of 500x for edge region washer front surface