

3D X-ray Computed Tomography Investigation of Fiber Damage and Microfibril Orientation in Compression and Injection Moulded Composites.

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ABSTRACT: The internal structure of short natural fiber reinforced composite were strongly linked to the mechanical properties of the composite. Therefore, it is required to anticipate the fiber damages and microfibrils orientation to ensure the composite strength through non destructive method. The focus of present investigation to ensure the shearing effect of natural fiber in twin screw extruder during mixing of PP matrix and bundle nature of pineapple fiber in compounding process develops microfibril and fiber damages in the specimen. The study was performed on 10% PALF/PP composite specimen developed in both compression and injection moulding process and examined under X ray microtomography and optical vision measuring system. The flaws, voids, curliness, microfibrils, fiber damages, orientation and fiber length measurement were detected in this method and compared both plastics manufacturing process and anticipated random distribution of fibers and orientation in composite product.

KEYWORDS: Fiber orientation, Microtomography, injection moulding process, compression moulding, composite defects,

I. INTRODUCTION

In the present scenario, the plastics industries on profitable motto producing complicated polymer parts in large volume with marginal cost. The advantage of using plastics in automobile industries is due to less weight, good aesthetics, require strength and low moisture absorption [2]. The synthetic fibers were reinforced with polymer to increase the mechanical properties of composite, whereas in recent years, natural fibers like sisal, coconut, and banana were reinforced with polymer for various application of bio-degradability [1]. The banana fiber reinforced with polymer exhibit an excellent mechanical property like tensile modulus, flexural strength and predicted banana fiber is stronger than sisal fiber [4]. The Felix reported that the addition of compatibilizing agent improves the adhesion of cellulose and polymer and estimated the elastics modulus and elongation at break for banana fiber in range 8-20GPa and 1-3.5 %, respectively. Thus the properties of natural fiber composite depend on the adhesion of fiber – matrix, orientation of fiber, aspect ratio of fiber [3],[5].

Sabu Thomas have analyzed the mechanical properties of short pineapple fiber reinforced in low density polyethylene (LDPE) of injection moulding process and examine the fiber – matrix adhesion and fiber orientation in scanning electron microscope (SEM) [5]. Mahesh Gupta developed a plague using injection moulding process and examines the glass fiber orientation on samples at four different locations. It was reported that the fibers were aligned near the surface in shell region and fiber orient transversely near the mid plane of core region. During the flow of polymer, fiber changes its orientation from horizontal to transverse due to melt front in gap wise divergence of thick section of part [6].

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The orientation of short glass fiber reinforced thermoplastics governs the strength, toughness and stiffness. The concentration of fibers in composites increases the strength and viscosity at lower shear rate with preferred orientation in radial direction of flow [7]. In the compression moulding process of composites, the orientation of liquid crystalline polymer LCP/PA66 blend exhibit a random orientation due to low viscosity nature of melt and were disperse uniformly in the composite. The micro fiber of random orientation has higher storage modulus and improves the mechanical properties of composites [8].

In this research, the study was performed on microfibril orientation and damages in fibers reinforced composites were examined in both X ray microtomography and vision measuring method. The specimen was developed for 10% PALF fiber reinforced with polypropylene in both compression and injection moulding process and examine the fiber damages and microfibrils development during shearing of fiber bundles in compounding process of twin screw extruder. The nature of fiber orientation was predicted using micro tomography and analysis the fiber length, fiber dispersion and void comparison on samples developed in both injection and compression moulding process.

In this paper section II describes the preparation of specimen in compression and injection moulding process and the specimen were examined under computed tomography and vision measuring device. In Section III describes the result and discussion of images and predicted the measured dimension of fiber after manufacturing process. The comparison of images and process were described. The Section IV presents the conclusion of this research investigation.

II. EXPERIMENTAL WORK

2.1 MATERIAL:

The injection grade polypropylene H110MA (REPOL) with melt flow rate of 11gm /10 min was used as a matrix and obtained from M/s IOCL, India. The pineapple leaf fibers were used as reinforcement and procured from M/s VIBRANT NATURE, Chennai, India.

2.2 METHODS:

2.2.1 FIBER PREPARATION:

The Pineapple fiber (PALF) were washed with water and dried in oven at a temperature 60°C for 6 hours and chopped manually to size less than 10 mm. The chopped fibers were sieved and obtained uniform sized free from moisture.

2.2.2 MELT BLENDING:

The PALF of 10% by weight was dried in oven to remove moisture and mixed with 90% of injection grade polypropylene for compounding process. The process was carried in twin screw Co-rotating extruder with capacity 5-30Kg/hr, BERSTOFF D-300 Hannover make. The temperatures of each zones of extruder barrel were gradually increased from 120°C to 200°C with drive speed of 150 rpm for uniform blend of fiber with polymer. The composite blend was drawn from nozzle in continuous form and fed into pelletizer for cutting into pellets.

2.2.3 SPECIMEN PREPARATION:

The pellets form of composite raw material was dried in the oven for 4 hours at 60°C to remove moisture content. The sample was prepared in two stages of process carried in two roll mill process and compression moulding process. The dried composites pellets were fed into two roll mill TRM-155 model of M/s Neoplast and were process at temperature of 200°C to obtain homogeneous mixed composite sheet from two contrary rotors, which was driven at different speed by separate motor.

2.2.3.1 COMPRESSION MOULDING PROCESS:

The preheated composite sheet was fed into an open cavity of compression moulding machine CBX150 with capacity of 150Tons and closed with top half of mould under low pressure of 35MPa and temperature 170°C. The specimen was developed in sheet form of size 250 x 125 x 3 mm shown in the Fig. 1. The bottom surface and top surface of 3mm thick specimen was shown in the Fig. 1 (a) and Fig. 1 (b) respectively, where short PALF fibers reinforced with Polypropylene pellets were mixed in two roll mill and on compression process developed the specimen, to study the orientation of short fiber in composites.

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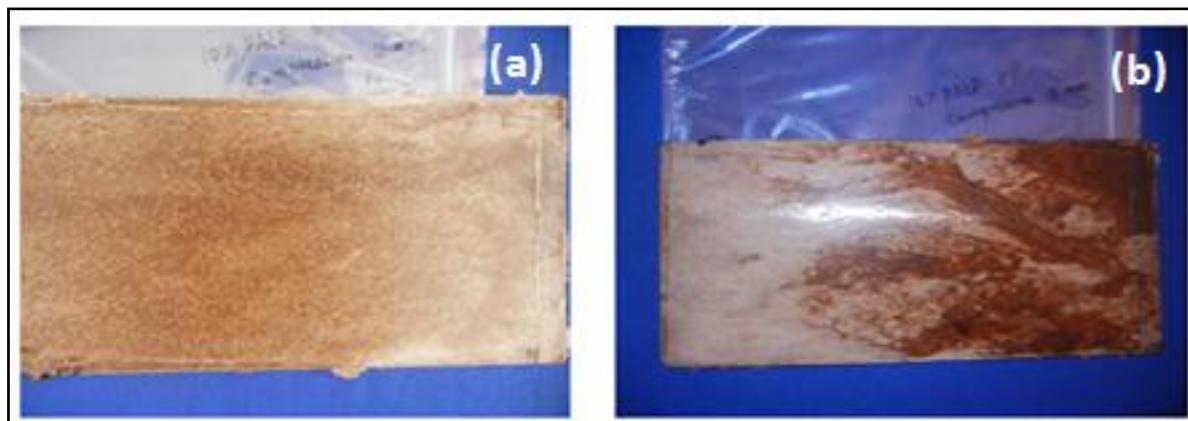


Fig. 1 : (a) Bottom surface of 3mm thick specimen, (b) Top surface of 3mm thick specimen.

The thin specimen was developed in compression moulding process, to study the orientation of PALF fiber in composites and shown in the Fig. 2 for 0.5mm thickness of composites. The Fig. 2 (a) shows thin film of PALF/PP composite sheet with distribution of fibers and the Fig.2 (b) shows the random distribution of micro-fibers viewed under optical microscopes – Vision measuring machine to study the fiber damages and microfibrils orientation in polymer composites.

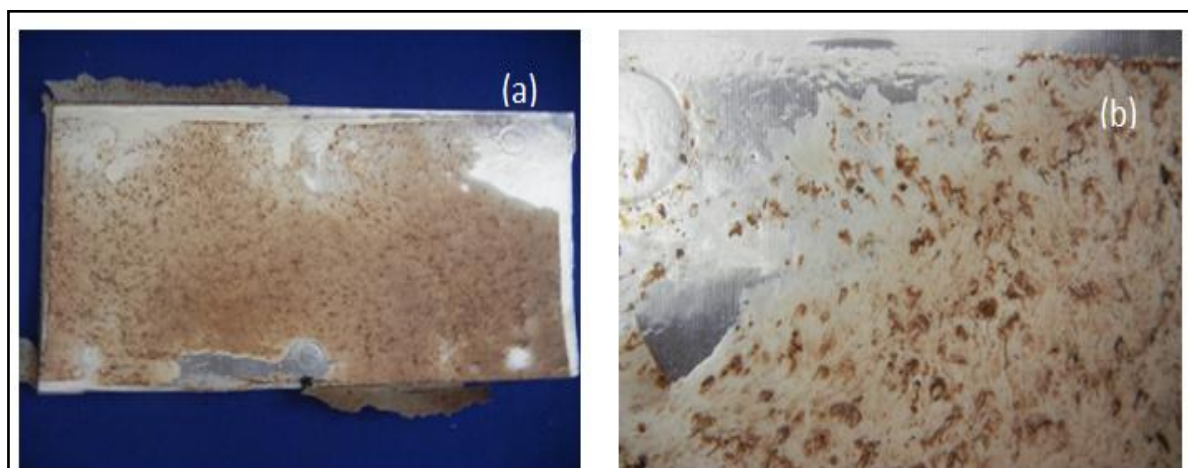


Fig. 2 : (a) Thin specimen of PALF/PP, (b) Random distribution of fibers in thin specimen

2.2.3.2 INJECTION MOULDING PROCESS:

The pellets were dried in oven for 5 hour to remove the moisture content at temperature 80°C and the specimen was moulded in Windsor SP-130, injection moulding machine. The temperature of the barrel were set to 160°C - 200°C and maintained mould temperature at 40°C with an injection pressure 50MPa, injection time 5sec, Holding time 12sec, cooling time 25 sec and holding pressure of 50MPa. The cut specimen was shown in the Fig. 3 to examine the orientation and surface were polished using buffing tool.

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Fig. 3: Cut sample from injection moulding process

2.3 MEASUREMENT OF FIBER ORIENTATION:

The orientation techniques were applied to study the dispersion of fibers in polymer composites and two methods such as optical vision measuring system and X ray micro tomography equipments. These methods were used to view the orientation of fibers and to predict the micro fiber breakages inside the composite moulded components. The specimen was cut across the cross section, to a size of 10x2x3mm shown in the Fig. 4 to examine.



Fig. 4: Cut sample for Micro-Tomography

2.3.1 OPTICAL MICROSCOPIC STUDY:

The geometry of the fiber and physical dimension of the component were determined by optical measuring device and the micro dimension of fiber were measured using CNC vision measuring system- EV3020. The optical images were digitized using Opus software to measure the physical dimension with an accuracy of 10 micron. The surface textures of specimen were optically viewed and digitized images to predict the orientation and studied the fiber dispersion of

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composite. The optical CNC vision measuring equipment was used to find the fiber length, diameter and curliness of fiber in a polymer composited.

2.3.2 X RAY MICROTOMOGRAPHY:

In this method, the internal structures of composite were inspected and fiber arrangement images in matrix were digitized to study the orientation. The porosity of 5micron can be predicted and visualize using non destructive testing techniques (D. Salaberge et.al., 2010). The General Electric and control solution- Germany, has developed X ray tube to focus micro and nano size with high power of 180KV of voxel size of 6 μ m. The capacity of nano focus tube at 180 Kv and micro focus tube at 240Kv provides a resolution of 0.5 μ m and 10 μ m respectively. (M. Bilewicz et. al. 2008, J. Kastner et. al. 2012). The composite samples were investigated at current 220 μ Amps and voltage 55KV with voxel resolution of 5.9 μ m and attain images through data acquisition. The reconstruction images of fiber arrangement were retrieved using inbuilt GE Phoenix datosx software and converted to 3D volumetric sample using GE Phoenix datosx acquisition shown in the Fig. 5 (a).

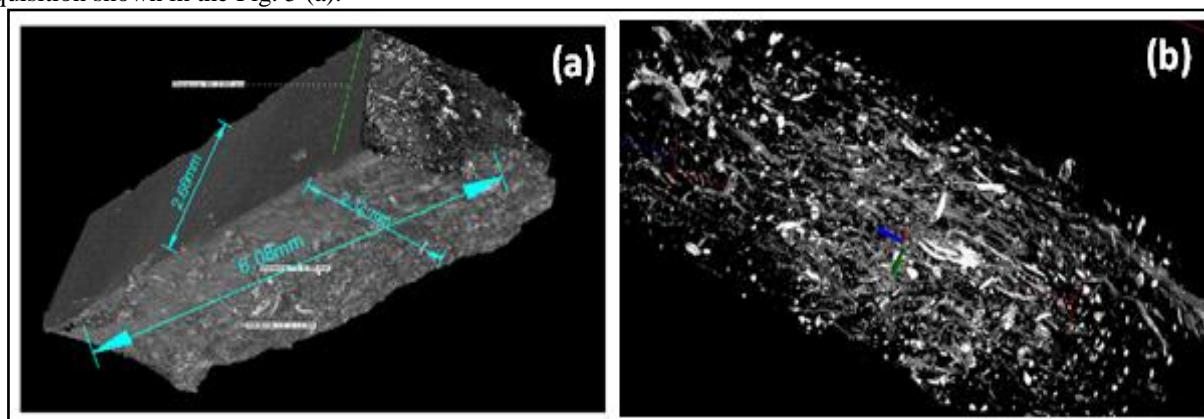


Fig. 5: (a) Sample size examine in Micro CT scan, (b) Micro CT image of PALF distribution in PP composite 3mm thick sample of compression moulding process.

The micro cracks, fiber length, fiber dispersion, fiber arrangements, interfacial voids, surface texture, and micro structures were determined through X-ray tomography (A.I.S. Brígida et. al, 2010, O. Brunke et. al. 2012). The datax software was used to remove image density of polymer from natural fiber and displayed grey scale of natural fibers, for analysing the geometric structure of fiber such as kink, curl, and aspect ratio and shown in the Fig. 5(b).

III. RESULT AND DISCUSSION

3.1 MICROTOMOGRAPHY IMAGE ANALYSES ON COMPOSITE:

The microtomography analysis was carried on the compression moulded PALF/PP composite specimen shown in the Fig. 5(a) and studied the micro fiber orientation and fiber damages during compounding process. The inbuilt Phoenix datosx software in computed tomography were applied to remove matrix from the composite and display only short fibers shown in the Fig. 5(b) and authenticates random orientation effects in fiber composite. The density of fiber and polymer are different, so the image techniques were used to highlight only high density fiber from polymer. The Fig. 6 (d) shows the 3D view of cut sample, where the three sectional planes were displayed for the sectional view of images. The Fig. 6(a) shows the sectional front view of cut specimen at specified position and magnified for analysis, found void, curling effect in fiber, and fiber damages.

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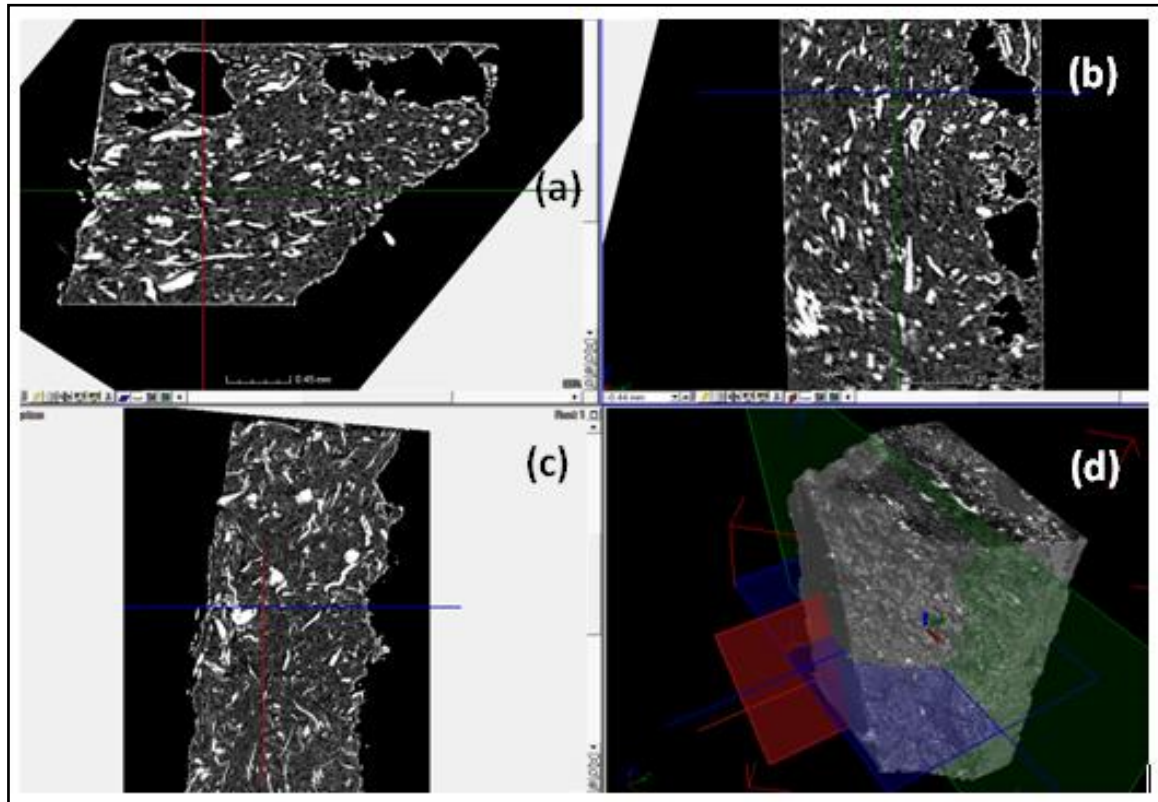


Fig. 6 : (a) Tomography image Top view, (b) Sectional image of Front View, (c) Sectional image of left side view, (d) 3D image of composite sample.

The Fig. 6(b) shows the sectional side view of sample at specific position and found the orientation of same fibers in another view. The void and curling effect are also present in composite, subsequently sectional view were analysed for various position of section. The Fig. 6(c) shows the sectional plan view of specimen at specified position and found microfibrils present in composites. The X ray tomography images have differentiated the fiber and polymer, by means of grey scales of colour and the images clearly illustrate the fiber aspect ratio, curliness and random dispersion in the composite.

3.2 VOIDS IN PALF/ PP COMPOSITE:

The image comparison on made between microtomography and optical vision measure machine and predicted the voids, microfiber, fiber geometry, moisture void and blow holes. The Fig. 7 (a) clearly shows the large number of voids occurs in compression moulding process and exhibit micro fibers in the sectional view. A similar investigation was done by P. J. Schilling et. al, 2005,[14] in microtomography for 26 micron pixel size and on reconstruction of sectional images, found the void location and its geometrical size. The area of specimen were zoomed to scale of 45micron and found natural fiber are deformed and kink in nature, which authenticates random orientation for natural fiber in polymer composites.

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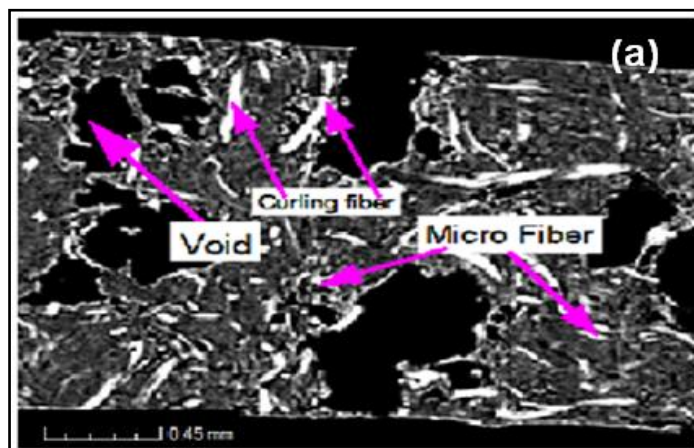
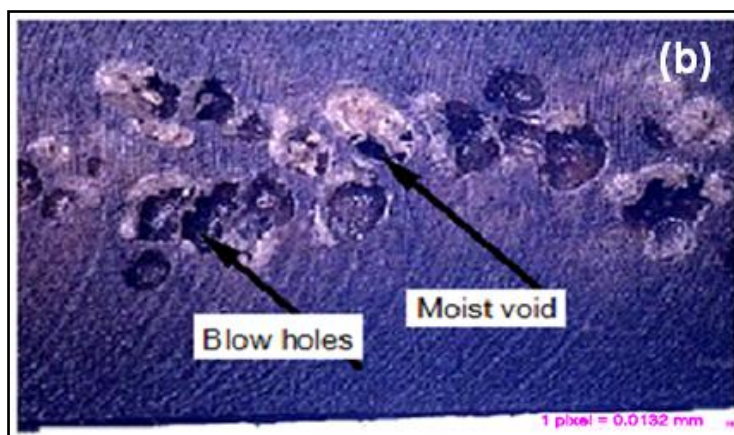


Fig. 7 (a) Tomography image of Void in compression moulded sample,

The Fig. 7 (b) shows the optical microscopic images of cut section sample of injection moulding process, clearly shows the macro void occur near the surface of the natural fiber due to presence of moist content. The interfacial stress between the fiber and matrix was weak because of no fiber treatment with compatibilizers, and blow holes happened due to moist gas entrap. The fiber arrangement and voids measurement in microtomography instrument has high accuracy compare to optical vision measuring machine.



7. (b) Optical microscopic image of moist void in injection moulded sample.

The tomography image of injection moulded sample shown in the Fig. 7(c) was found that the fibers were horizontally oriented along skin layer and randomly oriented in core region. The same observation was agreed and reported in the literature of S.Jayamol George 1995[5]. The geometrical dimension of natural fiber were inconsistent because fiber breakage were less and exhibit fiber damages and microfibrils. The fibers are found in curling, bending, splitting confirms random orientation. From the Fig. 7(c) long fibers length present near the wall were oriented in align along fluid flow and short fiber were oriented randomly due to curliness.

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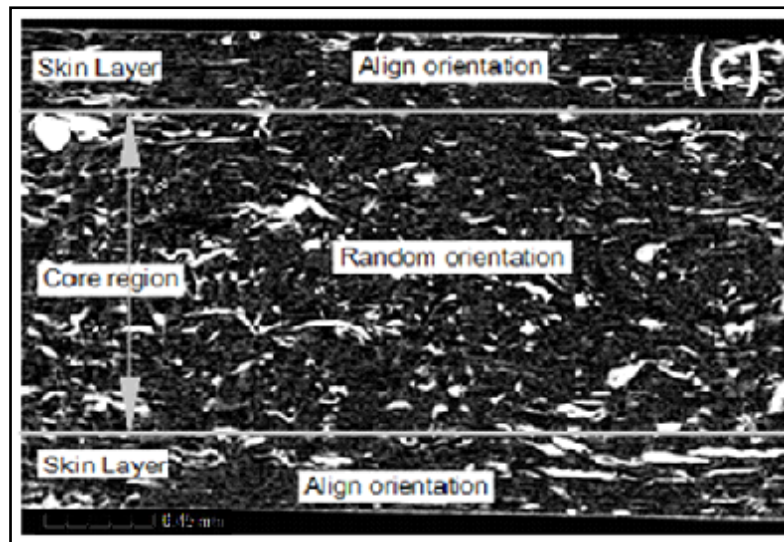


Fig. 7 (c) Tomography image of fiber orientation in injection moulding process.

The Fig. 8 shows the X-ray tomography image of injection mould sample of 10%PALF/PP composite and the fibers were arranged randomly in matrix. The 3D image of specimen shown in the Fig. 8 (a) clearly confirms the fiber lengths were inconsistent and bends unpredictably inside polymer composites. The fiber- fiber interactions and random distribution of fiber are found to resist the flow and form random orientation.

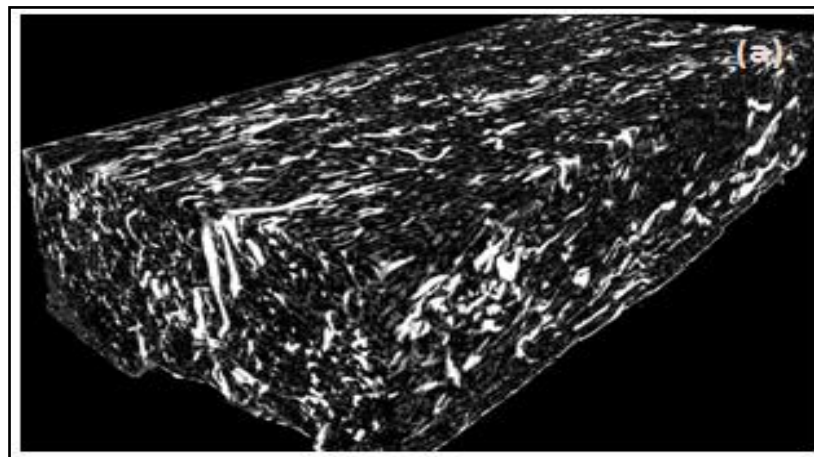


Fig. 8 (a), 3D Tomography image of 10% PALF/PP in injection moulded sample.

The Fig. 8 (b) clearly shows the voids at the interface of fibers and matrix, found less voids as compare to the measured sample of compression moulding process and was previously investigated in literature by Paul Schilling 2005 [14]. The fibers were found aligned along the boundary wall and transverse in core region, which was reported by Mahesh Gupta 1993[6]. The void availability in injection moulding sample was found to less compare to compression moulding sample and size of voids are found smaller at the interface of polymer and fiber edges.

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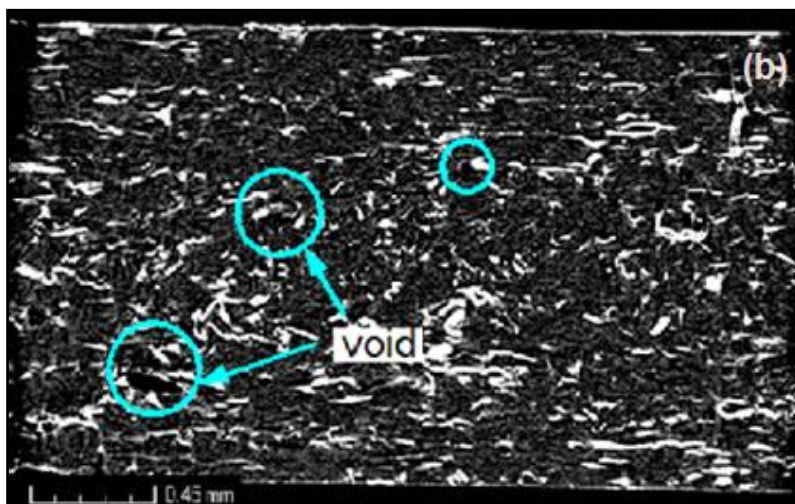


Fig. 8 (b) Tomography image of void in injection moulded sample.

From the Fig. 8 (c), the fiber bundles were distributed invariably across the section and were sheared along the direction of flow in injection moulded process. It was observed that the bundle fibers were damaged to microfibrils during the shearing composites in twin screw extruder and microfibrils are exhibited in the examined surface. The natural fibers are flexible that fiber bend and deform against the direction of polymer flow. It also observed that the fiber flow were resisted and varies along the skin layer to core region of composites, which already reported by S.Jayamol George 1995[5].

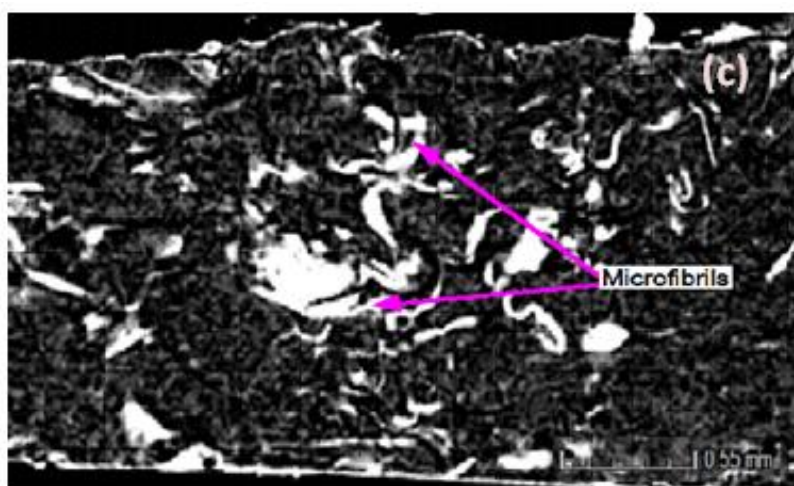


Fig. 8 (c) Tomography image of micro fibers in injection moulded sample

3.2.1 FIBER CLOGGING:

The dispersion of fiber in polymer composites depends on following parameters like pressure, temperature, inlet gate size, vent etc., and also the length, stiffness and flexibility of fiber influences the uniform dispersion in polymer composites [15]. From the Fig. 9 (a) it was observed that the fibers were clogging near the inlet gate, exhibit random distribution of smaller fiber less than gate size. This clogging enables decrease in flow rate against the freeze of

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molten polymer, results in incomplete filling of cavity. The Fig. 9 (b) clearly shows the short shot defect in composite, due to clogging of long fiber and stiffness nature, resulting in improper filling in cavity.



Fig. 9: (a) Fiber clogging, (b) Short shot

3.3 ORIENTATION OF FIBER IN MOULDING PROCESS:

The fiber distribution in polymer composite enhance the orientation of short fiber in a prefer pattern depends on the shearing rate of polymer flow and enable to improve the modulus of composites [6]. The optical microscopic image of composite confirms the orientation of PALF fiber was randomly distributed on surface of composites. The Fig. 10 (a) shows the surface images of compression moulded 3mm thick sample and was examined in optical vision measuring machine, which clearly depict random orientation of fibers on planar surface of boundary. The Fig. 10 (b) shows the macroscopic image of thin compression moulded sample was examined in optical vision measuring machine, authenticating random distribution of fibers.

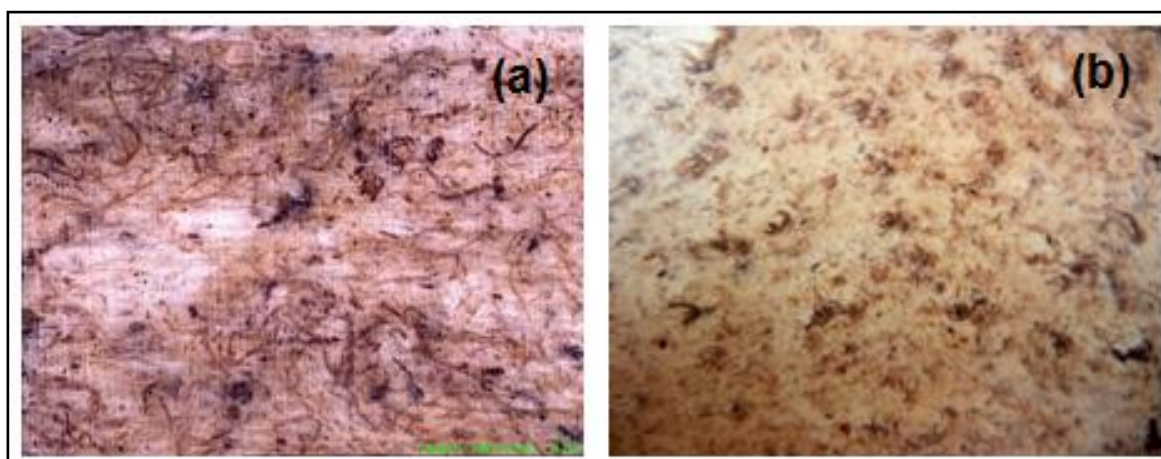


Fig. 10 : (a) Random distribution of fibers on Top surface of 3mm Thick sample of compression moulding process, (b) Optical microscopic view of random distribution of fibers exhibit on surface of thin sample of compression moulding process.

The investigation was further focused to study the micro fiber behaviour in thin specimen of compression moulding process and was observed from the Fig. 11(b) that there exhibit micro fibrils from single bundle fiber. This optical and X tomography images confirms that the micro fibrils, fiber damages and fiber bend were happens during compounding process due to shearing of fiber and matrix mix during co-rotation of twin screw extruder. The Fig. 11 shows the microscopic composites images of Xray tomography, Optical microscopy and Scan electron microscope and compared the image analysis and clearly shows the microfibrils orientation in composites. From the Fig. 11(a) and Fig. (b) infer image-differences between X ray tomography method and optical vision measuring method on

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compression moulded samples and observed fibers distribution and micro-fibrils orientation were random in polymer matrix of composite.

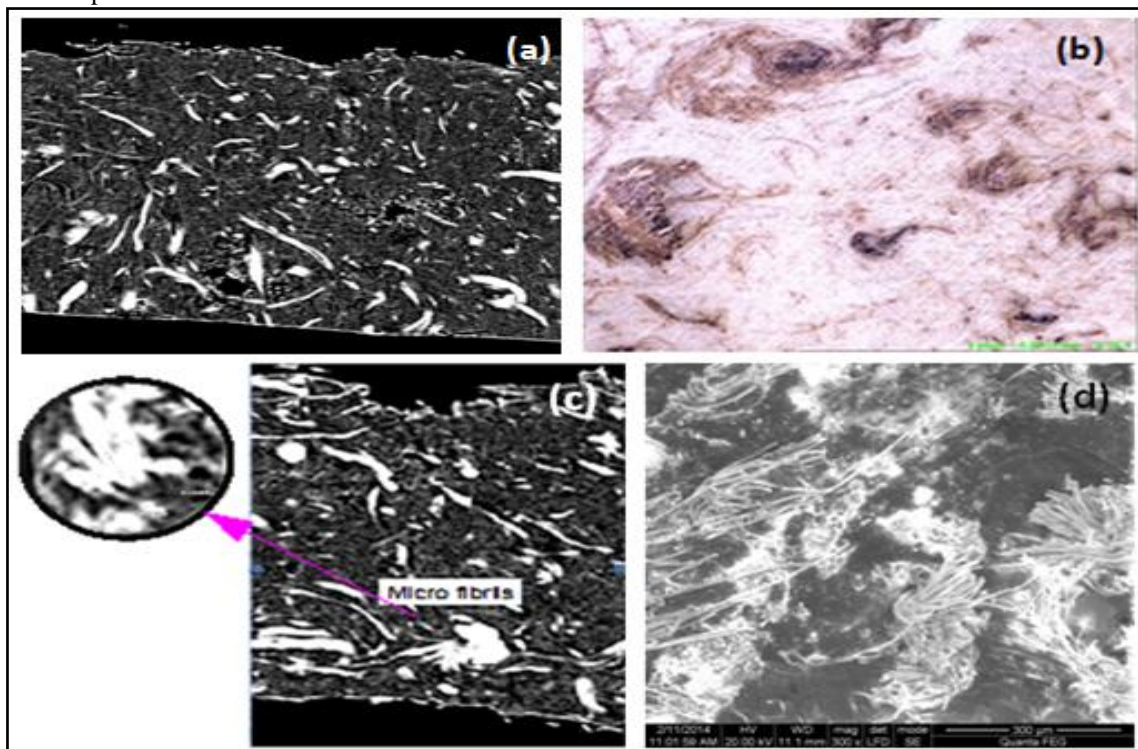


Fig. 11: (a) Micro Fibers orientation on thick sample of compression moulding process, (b) Micro fibrils exhibit in thin sample through vision measuring machine. (c) Tomography image of Micro- fibers exhibit after extrusion process in compression moulded sample, (d) SEM image of Micro- fibers exhibit after extrusion process in injection moulded sample.

The zoom scale image of Fig.10 (b) was further enlarged and shown in the Fig. 11 (b), which confirms microfibrils are flexible spread widely along direction of flow. It was also observed from the Fig. 11(c) and Fig. 8(c) that the micro-fibrils were found in the sliced section of X ray tomography and were compared with SEM images of Fig. 11(d) to authenticate micro-fibrils exhibit from single bundle fiber after compounding process.

3.3.1 FIBER DISTRIBUTION IN FLOW FRONT:

The flow front effect of the molten polymer decides the angular motion of fiber during filling of cavity and has no evidence for fiber motion in molten polymer. The Fig. 12(a) and Fig. 12(b) shows the fiber distribution along the flow field at end portion and inlet gate of injection moulded sample, respectively. It was clearly observed that the short fibers were distributed along flow field following the theory of flow, that viscous fluid motion has both rotational and translational effect. The velocities difference occur during mould filling, enables flow front and low density fibers are distributed along flow lines shown in the Fig. 12. The concentrations of fibers were found due to vorticity effect at low pressure area in the cavity form random distribution.

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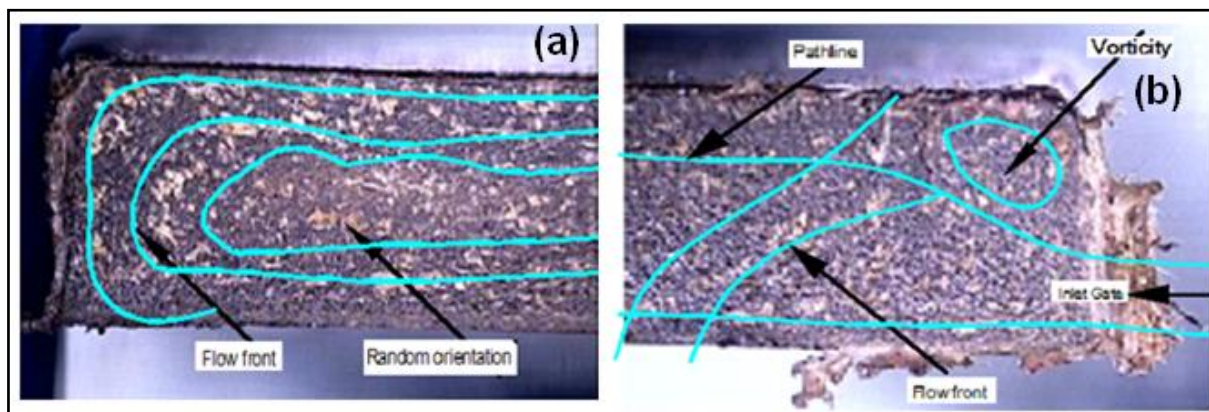


Fig.: 12 (a) Fiber orientation along flow front at end portion of injection moulded sample, (b) Random orientation of fiber near inlet gate of injection moulded sample

3.4 EFFECT OF FIBER LENGTH:

The X ray tomography method were used to measure and quantify the fiber length, void dimension, fiber agglomeration, internal damages were already reported by Paul J.Schilling 2005, [15] . From the Fig. 13, the length of fiber was found to be 0.5mm to 0.05mm and aspect ratio of fiber was non uniform resulting random orientation. It was also found that voids and fiber agglomeration in composite, which may leads to propagation of crack under loading condition. The tomography non destructive method of inspection has identified the defects, blow holes, fiber bend, fiber curliness, fiber sizes and orientation, confirms micro fibrils exhibit during compounding process and natural fibers are randomly oriented in polymer composite.

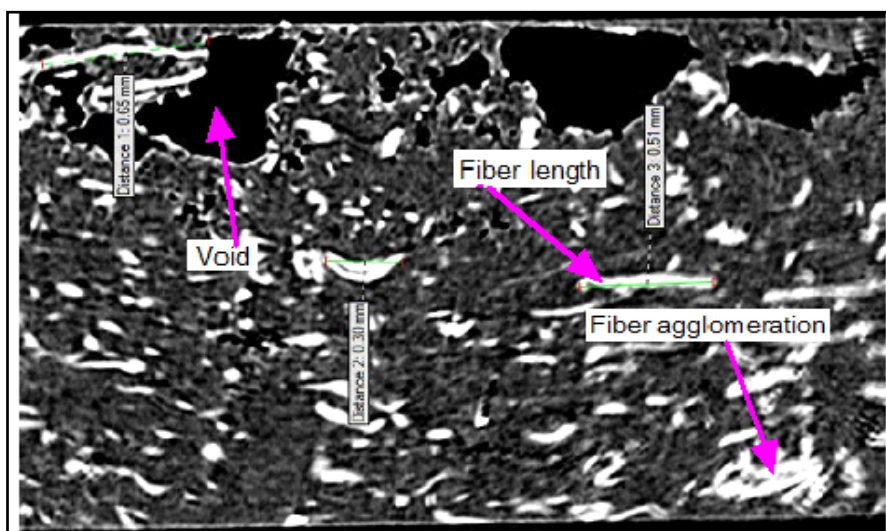


Fig. 13: Tomography image of fiber length and micro fiber determination in composite.

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IV. CONCLUSION

The image analysis was done in X ray micro tomography and vision measuring system for 10% PALF/PP composite samples in both compression and injection moulding process. The fibers were randomly distributed in the composite sample of compression process, whereas in injection moulding process, the fibers were aligned along wall and randomly distributed in the core region of composite. The micro fibrils were seen in the composite due to high shearing of fibers mix matrix during co-rotation of twin screw extruder in compounding process. The fiber lengths were reduced to 0.5mm to 0.05mm and natural fiber are found with curliness in composite through Xray tomography technique. The voids, fiber agglomeration and fiber orientation are detect through both X ray tomography and vision measure methods for both injection moulding and compression moulding process. The orientation of soft single PALF fiber exhibit microfiber during compounding process and micro voids are predicted by non-destructive method of X ray tomography.

V. ACKNOWLEDGEMENT

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