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Original Article

Energy behavior assessment of rice husk fibres reinforced polymer composite

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ABSTRACT

Natural fibre composites, possess characteristics that are beneficial towards the environment and ecosystem, completely biodegradable, renewable, offer strength and reliability of the material properties, and enhance economic development because of its ability in replacing synthetic composites. One of them refers to rice husk, which is a non-timber source that is easily available from agricultural wastes. Hence, rice husk fibres were selected as fibre-reinforced composites, while polypropylene as matrices in this research. Rice husk composite (RHC) specimens were produced via injection molding process. The composite composition was fixed at 35% of fibre content. The cycle tests were conducted to obtain cyclic properties by adhering to specifications outlined in ASTM D3479. Servo-hydraulic machines were used for five different stresses with constant amplitudes of $R = 0.1$, $R = 0.3$, and $R = 0.5$ of S75, S80, S85, S90, and S95 to determine the stress effects on energy dissipation and fatigue life of the material. The results indicated that there was increment in energy loss for the increasing fatigue life in every cycle. This shows that with the addition of natural fibres in composites, the material exhibited viscoelastic behaviour, in which energy loss was represented by matrix cracks and broken fibres.

1. Introduction

Over the past five decades, there has been a significant change in the industry in terms of use of materials. With the development in materials, the paradigm has shifted from heavy metals and homogenous to more flexible and versatile polymer matrix composites. To date, with rapid technology advancement, as well as clearer understanding of chemistry and physics aspects of polymer, there is potential for a combination of fibres to produce a wide range of materials called advanced composites [1]. The amazing stability and physical behaviour displayed by the composite materials make it ideal in engineering materials applications, as they are compatible with metals, particularly for use of structures [2].

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Many studies concerning mechanical testing have been extensively carried out by researchers to determine the optimal mechanical characteristics of composites. Hence, a range of studies has looked into the use of natural fibres as reinforcement in various types of polymer in recent years [3]. Many have reported that natural fibres have an added value advantage over metal structure and synthetic fibres because they are easily available, low in cost, low in density, have specific and acceptable strength, non-abrasiveness, good electrical resistance, good thermal properties, good acoustic properties, excellent broken resistance, ease of fibre surface formation, biodegradable, renewable, and emit lower CO_2 emission [4].

Most engineering components are exposed to repeated stresses. This failure is known as fatigue failure of the material or component. Fatigue failure refers to a process that causes damages to materials and structures under the magnitude of fluctuation loads that are lower than the static failure load [5]. Accumulated damage may result in gradual reduction of mechanical properties, such as strength and stiffness. It may cause crack growth and eventually become failed or broken. The failure process speed is controlled primarily by the magnitude of the fluctuation load or the deformation cycle [6], which is usually referred to as strain or stress range. One parameter that has an adverse impact on load is mean amplitude or load cycle peak, although this particular parameter is less significant than stress range.

Fractures in homogeneous materials can be trans-granular or inter-granular fractures. As for composite materials, cracks are usually limited to matrix material, as it is most vulnerable to less resistance condition. Matrix materials tend to fail in a fragile way, and cracking occurs quickly in little or no deformation of plastic [7]. Detection of cracks under unstable condition becomes a critical issue in material or component failure. Hence, this study investigated the failure behaviour of rice husk composite material through lost energy assessment. The specimen was tested using cyclic load with a constant R ratio and different stress values. Based on the structural analysis, disastrous failure in rice husk composite occurred when the interfacial fibre-matrix material system turned weak as shear fibres and fibres were pulled during stress and no stress conditions. This accumulated internal energy dissipation mechanism illustrates the damage that occurred.

2. Hysterical energy density as fatigue failure index

Most materials exhibit ideal elastic behaviour, even in very little tension values. When a material is under cyclical deformation, it is considered to experience an irreversible energy loss. There are various factors that lead to such loss, including irreversible transfer of mechanical energy into heat, growth of cracks and other defects, and micro plastic deformation of crystals, to name a few [8]. The definition of energy dissipation at each loading and unloading cycles states that the overall energy consists of elastic energy and plastic energy. Elastic energy is released after it is unloaded to zero load. Meanwhile, plastic energy is divided into two aspects: from shear state of matrix, and from covariance of matrix [9]. Based on the experimental outcomes, it is impossible to separate both parts of the plastic energy. Nevertheless, scientists have considered that this energy with heat dissipation is a small fraction of the energy. Most energy is used by elastic and plastic deformation, including the effect on temperature rise.

Energy dissipation is a popular phenomenon in studying the behaviour of materials and has long been an interest amongst scientists and engineers. When stress is applied to polymer, some energy is released by the movement of molecular chain - viscous flow. These effects are related to time (speed or frequency) and temperature, which depend on superposition temperature/principle. Energy dissipation is mainly due to the viscoelastic adhesion behaviour [10], while the actual quantitative effects of this dissipation mechanism have yet to be resolved. Some factors that affect different energy releases in fibre-reinforced composites are viscoelastic nature of matrix and/or fibre materials, damping due to interphase, damping due to damage, viscoplastic damping, and thermoelastic damping [11]. Material strength is directly dependent on energy dissipation. Hence, various efforts have been made to develop analytical methods and/or energy dissipation calculation.

Research findings show, the finite element methods combined with cell modelling successfully capable analysed plastic energy dissipation in particle-reinforced metal composites [12]. They discovered that energy dissipation in composite mainly depends on the amplitude of load, the volume fraction and inclusion modulus under cyclic load, and at the absence of interfacial debonding between inclusion and matrix. Bucknall and Smith proposed a multi-craze theory and discovered that nucleation and evolution of the craze led to more energy loss. This energy dissipation is contributed from the propagation of a craze to micro crack, consists a network of fibrils crossing the craze planes from which the network develops concentration stress by extracting new material from the active stress- plane [13]. Adams and Bacon developed a macro mechanical model for unidirectional fibre-reinforced composites. It states that the energy lost in unidirectional laminate is the amount of separate energy lost due to longitude stress, horizontal stress, and shear stress. As a result, specific damping capacities are defined as the loss energy ratio to overcome stored energy. Hysteresis experiment is a simple method that analyses the nature of polymer energy dissipation [14]. The area surrounded by hysteresis loop corresponds to energy release for each cycle.

In this paper, the variance between lost energy values in the first mechanical cycle had been assessed. The estimation of lost energy changes in the cycle number function was implemented. The test method for identifying the quality of new polymer behaviour based on hysteresis loop is presented in this study. The proposed model is based on the energy balance of control volume V of the fatigue test material. The first law of thermodynamics states the following: mechanical energy input (W) = dissipated thermal energy (Q) + variation of energy in the variation of internal energy (U) [15]. Referring to the fatigue cycle, the energy balance equation is as follows:

$$\Delta W = \Delta Q + \Delta U \tag{1}$$

Mechanical energy inputs are cycles during fatigue tests and are measured with hysteresis loop area. Part of the mechanical energy is released as heat to the surrounding. Generally, heat transfer mechanisms are conduction, convection, and radiation, in which one heat transfer mode may occur on the other depending on the condition and laboratory test materials. The variation of internal energy is a state variable that contributes to non-damaging (non-elastic) and damaging (in fatigue sense) of energy use in the material.

3. Methodology

3.1. Preparation of materials

The preparation of RH-PP pellets was carried out through the extrusion process with the addition of compatibiliser agents. In this study, 35 wt% filling of rice husk fibre composition was used as natural fibre in producing the RH-PP pellets. The RH fibre sized between 100 and 500 mm had been used as fibre content with Structol TR016 mixture as the compatibiliser agent. Compatibiliser agent serves as an effective binder and offers good adhesion between fibre and matrix. It is essential for polymer to have filler content with a uniform mix in maintaining or improving its physical properties [16]. Low molecular weight minimises viscosity during the process, thereby increasing the flow characteristics. PP with 0.90-0.91 gm/cm^3 gravity (supplied by Polypropylene Malaysia Sdn Bhd) was selected as polymer matrix as it is environmental-friendly and cost-effective, apart from adhering to the technical requirements.

In this study, RH-PP composites were prepared via injection molding technique at the SIRIM Green Laboratory, Shah Alam. Rice husk fibre was dried at 105 °C in an air-drying oven for 24 h to remove trapped moisture in 1–2% content, and later stored in sealed containers. In order to obtain a better mixture, RH was mixed with PP using a tumbler mixer. Next, the compound was mixed with twin-screw extruders at 160 °C and 190 °C for feed zone and die zone, respectively. The twin-screw extruders refer to screws that rotate together and have 33 L/D. With 100 rpm of screw speed, the compound was extracted and pelletized. The pellets were stored in closed containers and dried for about 3–4 hours prior to pouring.

3.2. Preparation of specimen

After the extrusion process, the pellet was injected using 60-ton Haiti MA600 II/130 (see Fig. 1) machine into the form illustrated in Fig. 2 based on ASTM638-03 specification. The experiment was conducted at Universiti Tenaga Nasional laboratory located in Selangor, Malaysia. Process parameters (e.g. injection pressure and speed, mold and liquid temperature, cooling and holding time, and holding pressure) can affect mechanical properties [17]. The temperature used for injection mold samples was 175 °C for both feed and die zones. The samples were injected at injection pressure of 45 kg/m^2 with 10 second cooling period.

3.3. Tensile test

Tension test was carried out by adhering to ASTM D638-3 standard specifications using universal testing machine (Instron 3365), as illustrated in Fig. 3. The dogbone-shaped geometric specimens were tested at a cross speed of 5 mm/min to maintain the continuous stress rate at specimen gauge length of 80 mm. Additionally, extensometer devices were used to measure elongation during testing.

Fig. 1 – 60-tonne injection molding machine.

Fig. 2 – Test specimen.

Table 1 – Mechanical properties for rice husk composite.

Properties	Value (MPa)
Ultimate tensile stress, σ_u	25
Yield stress, σ_y	23
Young modulus, E	686

3.4. Cyclic test

The cyclic test was conducted on Shimadzu Servopulser EHF-E test machine (see Fig. 4) under load control mode. The constant amplitude load used was sinusoidal-shaped in the frequency value of 20 Hz. Based on ASTM D3479 / D3479 M standards, the specimens were tested at least at five stress levels (e.g. 95%, 90%, 85%, 80%, and 75% ultimate strength) to determine the fatigue stress-life diagram. This composite was tested in tension-tension mode (at R = 0.1, 0.3 and 0.5 stress ratio).

4. Results and discussion

The bio-composite polypropylene matrix, which was reinforced with rice husks, was tested in static and cyclic conditions. Table 1 presents some properties of the materials, such as tensile strength, tensile modulus, and yield strength.

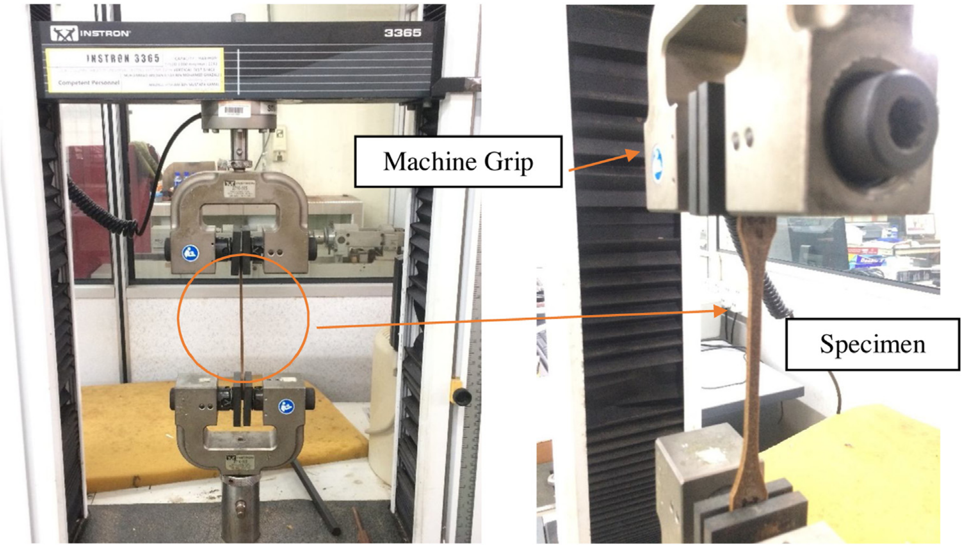


Fig. 3 – Tensile machine, Instron 3365.

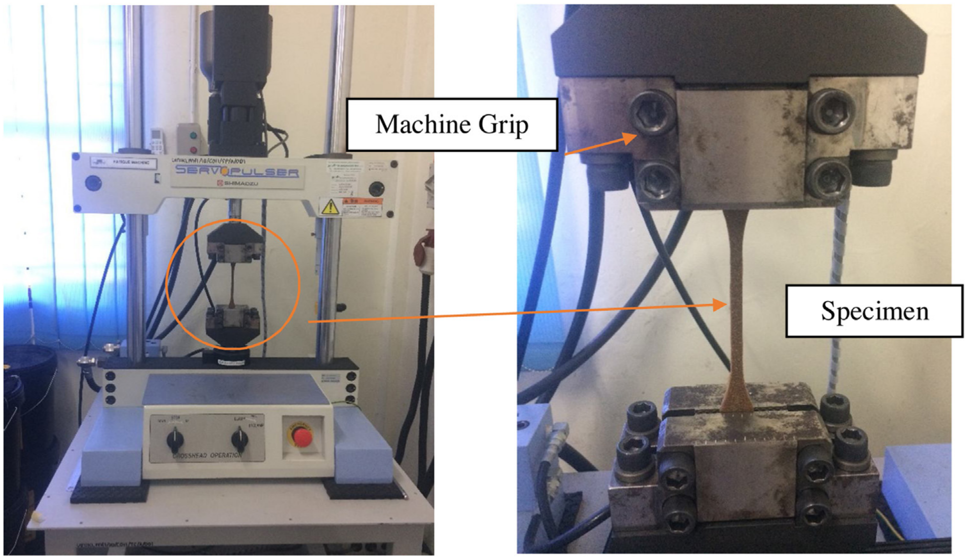


Fig. 4 – Cyclic test, Servopulser EHF-E.

These data were required for cyclic testing to determine the fatigue life of composite materials. Fatigue test was performed at $R = 0.1$, 0.3 and 0.5 stress ratio and 20 Hz for varying loads. The fatigue life values obtained are specified in Figs. 5–7, where this stress-life method is implemented to demonstrate a fatigue failure analysis based on the relationship between stress and fatigue life. S-N curve revealed the results of fatigue experiment. The Figs. 5–7 illustrates that the relative fatigue load, which is the maximum stress during recurring load, was represented by the fatigue life cycle logarithm at complete failure of each stress. The relationship between these two parameters is used in assessing the fatigue life of the material. Power-law regression equation based on Basquins' approach, $S = A\left(N_f\right)^B$ were determined for each of R-ratio, where S is the stress applied, N_f is the number of cycles to failure, A and B presents material fatigue parameters. The curve shows that the fatigue life of rice husk composite decreased upon increment in stress level for different R-ratio. From Figs. 5–7, a smaller value of material fatigue strength coefficient B (−0.042, −0.113 and −0.155) indicates a steeper slope of the S-N curve and thus reflect to faster fatigue strength degradation [18]. Like all composite materials, fatigue degradation in rice husk composite quantified as the loss of modulu/strength in the applied loading direction [19]. By using the curve technique for S-N curve, it was found that the R-squared curve indicated good correlation [20] values of $R^2 = 0.86$ for $R = 0.1$, $R^2 = 0.93$ for $R = 0.3$, and $R^2 = 0.95$ for $R = 0.5$, where R^2 statistical parameters showed the extent of the best fit curve.

Composite failure or particle reinforced composites in cyclic stress is usually preceded by the accumulation of various internal damage types [21]. During the damage process in the composite materials, the phenomena were initiated

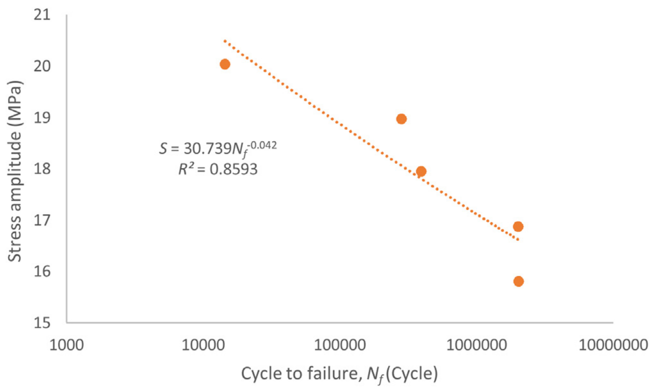


Fig. 5 – Tension-tension fatigue test for R = 0.1.

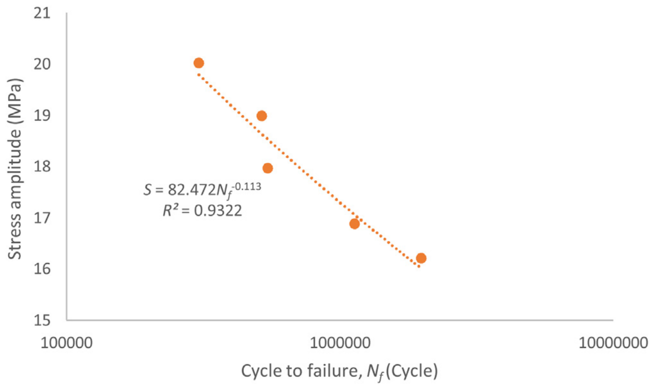


Fig. 6 – Tension-tension fatigue test for R = 0.3.

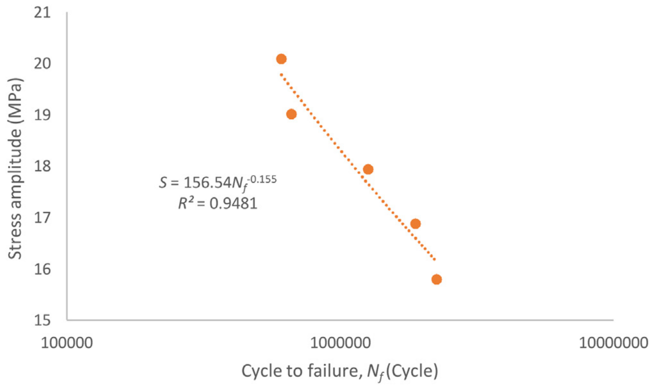


Fig. 7 – Tension-tension fatigue test for R = 0.5.

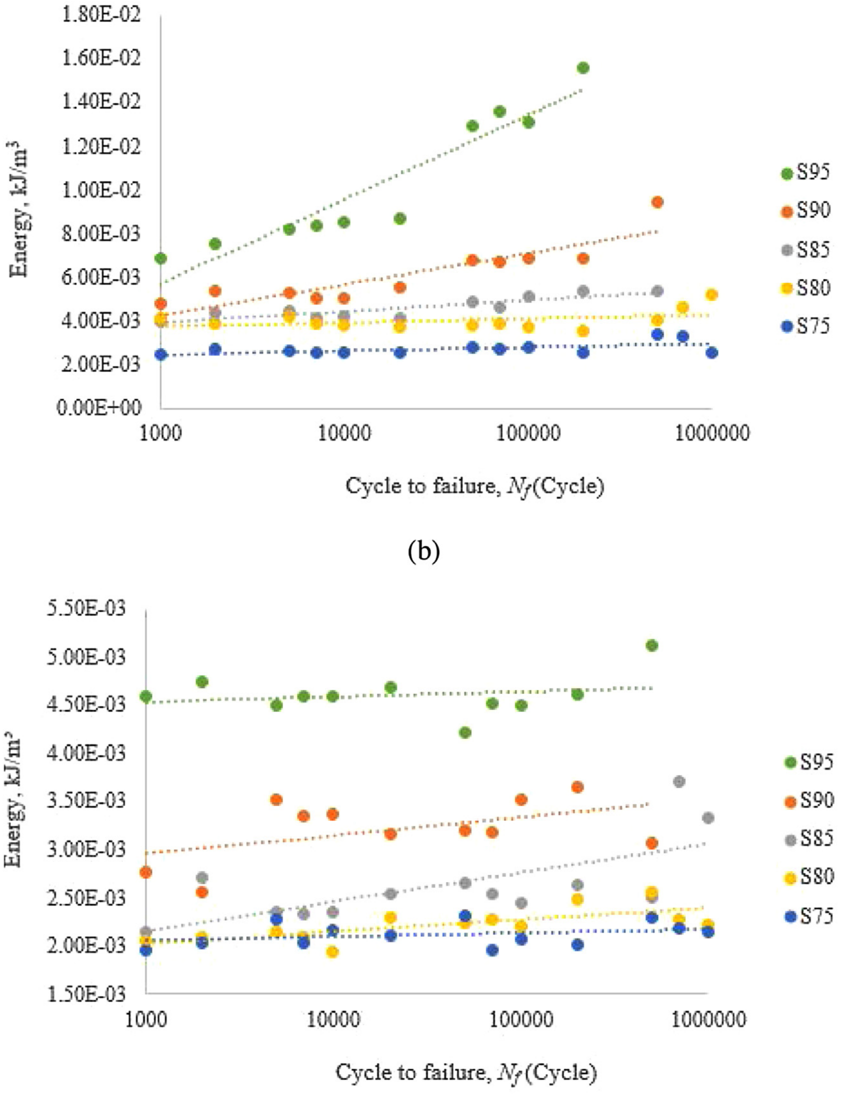
by formation and growth of micro-cracking or voids, clustering, coalescence, formation and growth of early cracking, as well as dispersion of one of the cracks until failure of the whole specimen [22]. These failure measures relied on the type of consolidation or particle (inclusion), and on the interface between inclusion and matrix. The effects differed depending on the applied load at the composite material. At the initial stage of stress, the debonding process of matrix interface occurred due to existence of weak inclusion matrix interface [23].

The load-sharing concept between matrix and amplifier constituents (fibres) is important in order to comprehend the mechanical behaviour of the composites. In composite materials, fibres are regularly surrounded by matrix materials. The external loads (force) applied to the material composites are partly borne by matrix and partly by reinforcement. In general, the external loads are directly charged to the matrix. Next, some loads are transferred to the fibres, while some are transferred to fibres through their end surface [24]. Under cyclic loading conditions, composite failure due to three phases (rapid stiffness loss, followed by slow decrease in stiffness and finally, a complete failure) [25]. At the first, matrix deformation start with crack propagation until load is completely transferred to the fibre length (stage 1). In stage 2, the fibres are in a state of strain, which slowly forming the cracks grow slower than before and the matrix debonds steadily from the fibers. Finally, catastrophic failure of the specimen occurred when the cracks propagate rapidly due to fibres breakage (stage 3).

Nonetheless, in the short fibres composition, the used fibre length was not too long, hence the occurrence of many stress transfers across the whole cylinder surface. Therefore, both ends of the fibres, as well as physical external cylinder surfaces, play an essential role in transferring matrix loads to the fibres. When the fibres are short, the similar tension condition will no longer occur under axial loads since the stresses in this phase tend to move towards the end of the fibres [26]. This means; the average stress in the matrix must be higher than that for longer fibre. Thus, lower stress that was applied in the fibre and average stress applied higher in the matrix affected the two main material behaviour; stiffness and strength of the composite, since the matrix was generally weaker and smaller than the fibres.

During fatigue failure, specimens were under recurring stresses, in which the stresses were lower than the materials required to cause failure. This trend signified increment in cycle energy for all stress levels (S_{75}, S_{80}, S_{85}, S_{90}, and S_{95}) until they were completely broken. The interaction between matrix and fibres is significant, especially for delivery of loads that is ultimately known as composite resistance. When a specimen is loaded, the load is delivered along the fibre axis, which is pushed towards both longitudinal and horizontal directions, hence known as response to dissipation source [27]. Based on the reaction, the weak bond between fibre and matrix gave higher hysteresis value, which indicated the relative movement of fibres. Fig. 8 shows that the highest energy dissipation occurred at stress S_{95} (the highest gradient), followed by stresses S_{90}, S_{85}, S_{80} and S_{75}, whereby the gradient obtained was more horizontal at S_{75}. This indicated that the released energy decreased due to the capability of the applied stress [28], in which S_{75} was lower to break the bond between the fibres and the matrix, in comparison to the higher stress, S_{95}. Therefore, at S_{95} stress state, the breakdown of bonds between fibres and matrix was abundant, resulting in higher energy release and the highest gradient [29].

At the beginning of fatigue failure, fibre damage occurred when the local stresses exceeded the weakest fibre strength, which led to the focus of shear stress at the fibre-matrix interface that was located close to the broken fibre edge [30]. The interface area served as stress convergent for longitudinal tension stress, which exceeded the matrix broken stress and caused horizontal cracks in the matrix. In fatigue failure, first, the crack spread randomly and slowly grew under repetitive stress action until complete failure. At this phase, the propa-



(b)

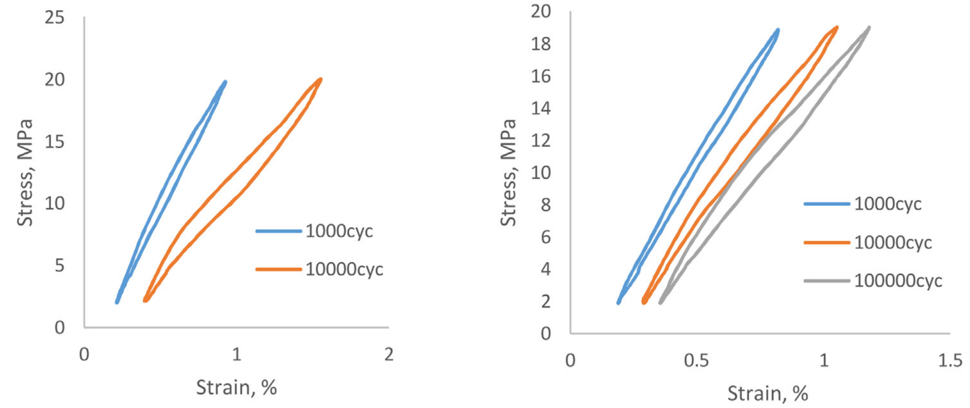
(c)

Fig. 8 – Energy dissipated versus the cycle to failure N_f of cycles for all stress level, (a) R = 0.1, (b) R = 0.3, (c) R = 0.5.

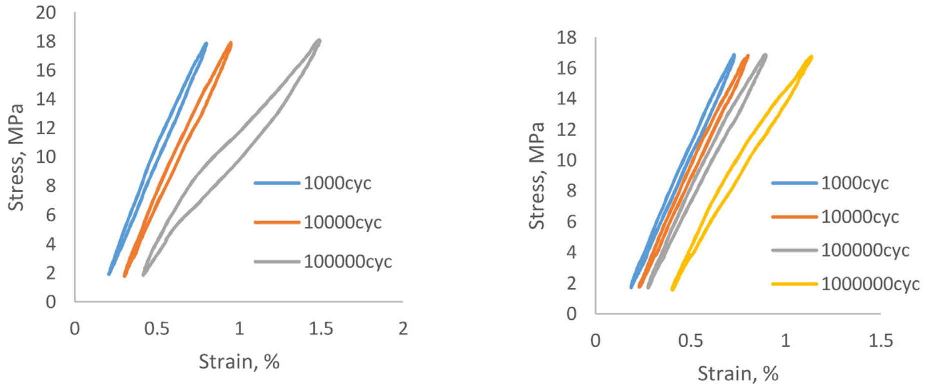
gation of the matrix crack failed due to the severe shear stress at the end of the crack. In the next phase, the cracks developed and grew until the final failure. Energy dissipation was determined by the progressive failure process in the matrix cracks with the related interfacial shear failures.

One of the analyses of damage phenomenon mechanism in the fibre-matrix material system seeks to comprehend energy dissipation in fatigue failure. This is important to reckon the behaviours of the material, as well as to open up new opportunities for design of tough, high-durability, and impact-resistant material systems with a large spectrum of industrial applications. The result for basic quantitative characterisation of intrinsic energy dissipation is presented. In the stress-strain diagrams (see Figs. 9–11), energy dissipation had been identified as an area covered by hysteresis loop [31]. In determining the lost energy for any maximum shear stress applied, the loading or unloading cycle was assumed as the area between the loading or unloading curves that represents the density of lost energy [32]. Figs. 9–11 clearly display a significant variance in energy dissipation during each cycle under the recurring load. By comparing energy dissipation during certain cycles in fatigue experiments, it is obvious that the loop area increases for each load level in the final phase of the test prior to failure. This refers to the structural instability caused by debonding fibres and cracking.

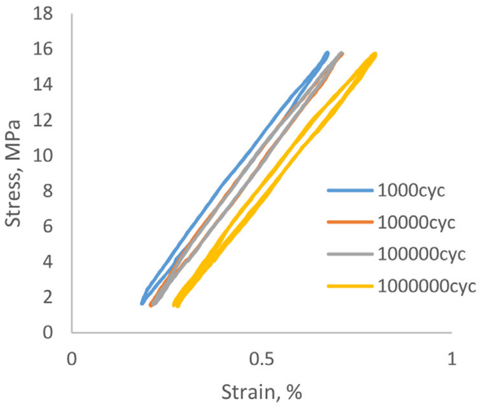
In metal, fatigue failures are closely linked to plasticity cycles, in which movement system and slip dislocation occur. Due to environmental impact and plane stress state, the beginning of fatigue failure often occurs near the metal surface. Fatigue failure dissipation in metal refers to a single crack. The fatigue failure of composite materials differs from the fatigue failure of metal. The simultaneous development of multiple cracks in composite materials causes difficulty in assessing fatigue damage based solely on single cracks. In addition, fatigue failure of composite materials depends on various damage mechanisms, such as breakage, matrix crack-



(a) *S*95 (b) *S*90



(c) *S*85 (d) *S*80

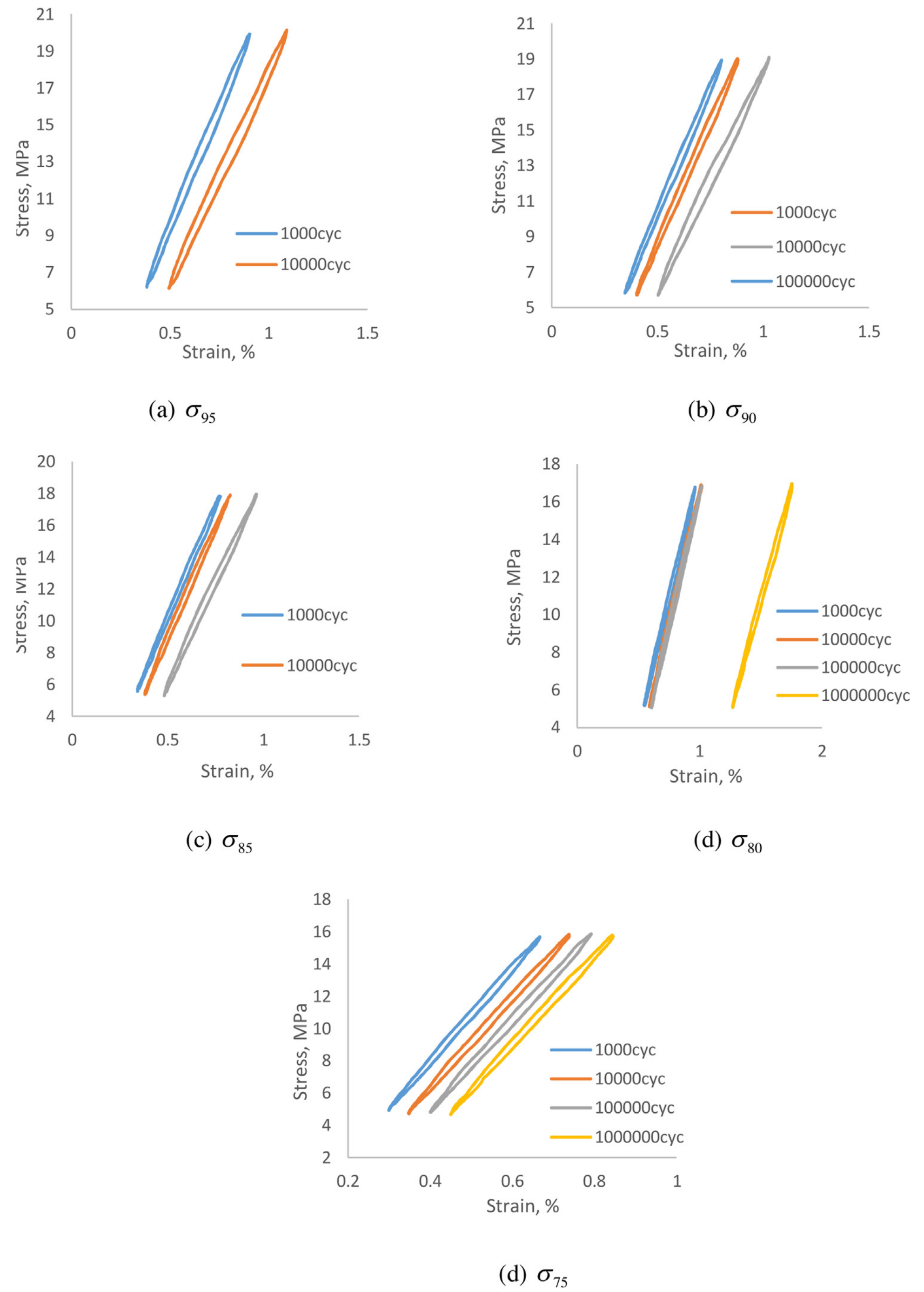


(e) *S*75

Fig. 9 – Energy dissipated for R = 0.1.

ing, delamination, and debonding [33]. This combination of damage mechanisms may affect the strength and stiffness of the material.

Fig. 12 illustrates the cracked surface of SEM image after the fatigue test. The energy dissipation mechanism in the fibre matrix material system consists of fibre breaks and failure, sliding friction on fibre matrix interface during fibre pull-out, and matrix changes [34]. The most interesting mechanism for energy dissipation in the fibre-matrix material system is associated with shear fibres and fibre pull-out. This mechanism has difficulty in exhibiting non-accumulative net-cracking fibre-matrix material throughout its cross-section, because it is only represented by some failures that exist in the structure of the component. Such fibre-matrix material systems allow



(a) σ_{95} (b) σ_{90}

(c) σ_{85} (d) σ_{80}

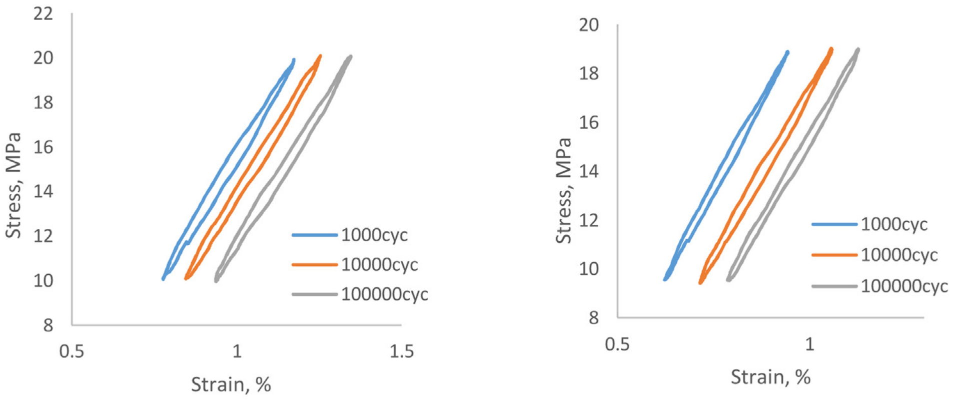
(d) σ_{75}

Fig. 10 – Energy dissipated for R = 0.3.

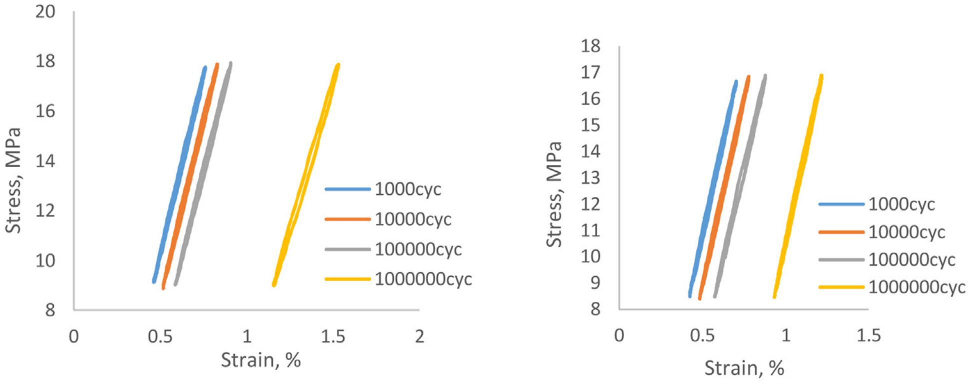
crack deflection on the fibre-matrix interface, and may cause fibre attachment phenomenon that prevents cracking from becoming a full failure. At the end of the failure, this material system exhibited many fibre pull-outs in several composite parts. The fibre pull-out length and the intrinsic strength control the amount of energy loss during the pull-out process.

5. Conclusion

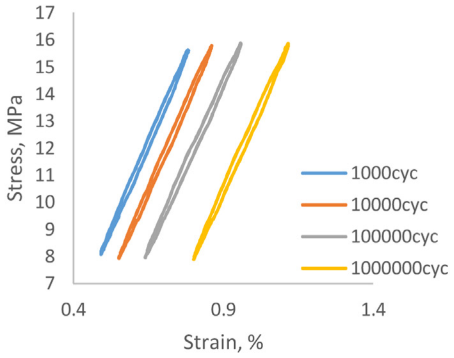
In this study, the composite of natural fibres, which refers to reinforcement of rice husk with polypropylene, had been assessed to determine the released energy value experienced by the cyclical load that was imposed on it. The released



(a) σ_{95} (b) σ_{90}



(c) σ_{85} (d) σ_{80}



(e) σ_{75}

Fig. 11 – Energy dissipated for $R = 0.5$.

energy value was stable after 1000 cycles. A correlation between dissipation energy value and mechanical behaviour of the composite was observed for the fatigue life of the whole material. The emitted energy increased in value upon increment in fatigue life. The addition of energy value is illustrated in the hysterical diagram plotted for each variance of R-value and load, wherein the space covered with hysteresis loops increased when the specimen approached complete failure. The released energy value was the highest for $R = 0.1$, which ranged between 5 kJ/m^3 and 30 kJ/m^3, whereas $R = 0.3$ for 2 kJ/m^3–16 kJ/m^3, and $R = 0.5$ for 2 kJ/m^3–5 kJ/m^3. The variance between the lost energy values in the cycle indicated fibres and polymer bonding strength, as well as the fluctuating internal stresses in the composites.

Based on the structural analysis, catastrophic failure in rice husk composite occurred when the interfacial fibres-matrix materials system turned weak due to fibre sliding and fibre pull-out during loading/unloading process. Upon failure, these

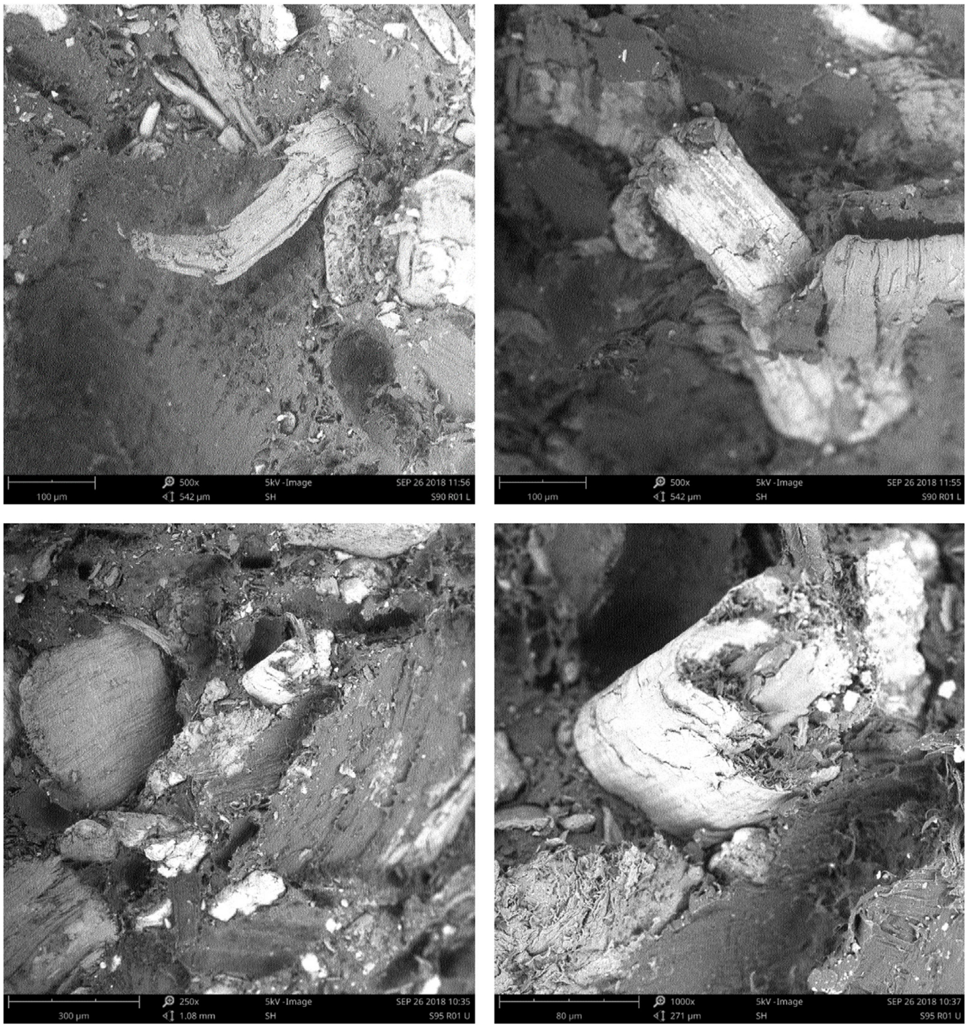


Fig. 12 – SEM image of fatigue test fracture surface of rice husk composite.

material systems exhibited extensive fibre pull-out at multiple sites. The length of fibre pull-out and the intrinsic strength of the fibre dictated the amount of energy being dissipated during the fibre pull-out process. This accumulative internal energy dissipation mechanism describes the damage that had taken place.

The mechanism of composite fatigue failure consisted of several interaction processes between matrix and fibres in load transmissions, which is known as composite resistance. When the specimen is loaded, the load is delivered along the fibre axis, which is pushed towards longitudinal and horizontal directions. This is the reaction to the energy dissipation source.

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