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Strain-Rate Sensitivity of Fiber-Reinforced Polymer Matrix

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Abstract

This paper evaluates the strain-rate sensitivity of fiber-reinforced polymer matrix. The methodology involved extracting fiber from plantain pseudostem, prepared and treated with nine different liquids; acetone, acetylation, glycerol, methanol, mercerization (NaOH), Methyl ethyl ketone peroxide (MEKP), hydrogen peroxide, potassium permanganate, and phosphoric acid. The fiber was prepared with the objective of molding cylindrical shape for mechanical test samples with continuous and unidirectional aligned fibers. Contact angle measurement was carried out in all the treated and untreated samples using two different probe liquid; water and glycerol. Matlab software tools were used in the mathematical analysis of the data generated from the experiments. Strain rate was calculated from adhesive energy- tensile stress model and used to determine the strain-rate sensitivity index, m for treated plantain fiber-reinforced polyester matrix. The low values of the strain rate suggest that the fiber sliding against the polymer matrix to which it was bonded was infinitesimally low and would not cause noticeable changes in their bonding between fiber and matrix. Since the strain rate sensitivity index in this study is less than 0.30, it can be concluded that the reinforced plastic is relaxed and so would not be adversely subjected to stress build up at the interface between the fiber and the matrix. This work shows that the use of adhesive energy-stress concept to determine the strain rate is valid since the values of strain rate indices calculated are comparable with values reported in the literature.

Keywords: strain rate; strain-rate sensitivity index, viscoelasticity, adhesive energy, tensile stress; composite

1. Introduction

The ability of certain polycrystalline materials to achieve large elongations in a tensile test without necking prior to failure describes the concept of superplasticity (Smolej et al., 2009). These elongations can be up to 1000% and in some cases even more. The superplastic forming (SPF) of aluminum-alloy sheets has been commercially established for more than 30 years. In general three conditions are needed to attain the SPF of material: the grain size should be very fine and stable (< 10μm), the flow stress must be low compared with those of conventional materials, and the strain-rate sensitivity index values m must be in the range 0.4 to 0.8 (Smolej et al., 2009). The strain-rate sensitivity index is considered to be the most important parameter that characterizes superplastic deformation. Viscoelastic phenomena in thermoplastics are of considerable importance in the design process in applications where the material is subject to loading at constant level of deformation for a prolonged period of time, such as filaments in tension and seals in compression. The strain-rate sensitivity index is then indicative of the degree to which the load will have diminished over time period and is dependent upon the molecular structure of the particular thermoplastic and the temperature of the environment (Gobble and Wolff, 1993). In this instance, the magnitude of a material's index value would be indicative of the performance characteristics of an *in situ* structure.

Time dependent properties are also of interest in the forming of thermoplastics where the material is subject to incremental deformation for a short period of time. In this case, the strain-rate sensitivity index is a measure of the change in dynamic stiffness with variation in strain rate and it is indicative of the deformation characteristics of the bulk material (Gobble and Wolff, 1993). In thermoplastics, the strain-rate sensitivity effect is manifested in the strain-rate dependence of the elastic modulus of the material loaded in tension. At low strain rate, the molecular chains have sufficient time to adjust to the imposed stress and the modulus value is lower than would be the case for the material loaded at high strain rate (Gobble and Wolff, 1993). Chandra and Roy (1987) reported that virtually all

thermoplastics exhibit some degree of room temperature strain-rate sensitivity. The stress-strain rate behavior of metals at low temperatures was reported by Backofen et al. (1964) to obey the power law relation:

$$\sigma = [k\acute{\varepsilon}^{m}]_{\varepsilon, T} \quad (1)$$

Where: σ is the flow stress; ἑ is the strain rate; m is strain-rate sensitivity index ($0 < m < 1$) which is a function of the forming parameters, such as strain rate and the temperature and is connected with microstructural characteristics; k is a dynamic modulus.

Equation 1 may also be related to common models for linear viscoelastic materials. Leterrier and G'Sell, (1967) reported on the relationship between the strain-rate sensitivity index and temperature in thermosetting polyurethane resin and found that the index m increased with increasing temperature below the glass transition temperature at which point further temperature increase resulted in decrease in m-value.

In a structural member under load, it is important to have an indication of the degree to which the load would have diminished over time. This is measured by a parameter, m, called strain rate sensitivity index, given in equation 2, at constant strain, ϵ, and temperature, T (Hart, 1967; Gobble and Wolff, 1993).

$$m = [\partial \ln(\sigma)]/[\partial \ln(\dot{\epsilon})]_{\epsilon,T} \quad (2)$$

Where: σ is the stress and è is the strain rate and it is assumed that the conditions approximate a steady-state process. The strain-rate sensitivity index can in principle be determined by stress-relaxation testing. The strain rate sensitivity index, m, which is indicative of the degree to which the load would have diminished or increased over time is dependent upon the molecular structure of the particular thermoplastic and the environmental temperature. In this instance, the magnitude of a material's index value would be indicative of the performance characteristics of an *in situ* structure. In thermoplastics, strain rate sensitivity effect is manifested as the strain rate dependence of the elastic modulus of the material loaded in tension. When thermoplastics are loaded at low strain rate, the molecular chains have sufficient time to adjust to the imposed stress and the modulus value is lower than would be the case for the same material loaded at a higher rate. For a relaxed viscoelastic material m = 0 where generally $0 < m < 1.0$.

This paper ventures into the determination of the strain-rate sensitivity in fiber-reinforced thermoplastics under tensile loading. Plantain fibers were used to reinforce the viscoelastic material used in this study. Stress at failure of the composite was determined and used for analysis. This study is important in determining the integrity of the fiber in the matrix under loading, whether the bond between the matrix and the fiber is strong enough to resist failure. High strain rate may suggest imminent material failure.

2.0 Methodology

2.1 Strain Rate Model

Aran, (1979) described numerous methods that have been utilized for determination of strain-rate sensitivity index including stress-relaxation testing. Hedworth and Stowell, (1971) cautioned against these methods as exhibiting little correlation to the actual physical processes. Stress was generally regarded as the preferred method for strain-rate sensitivity index determination when the objective of the investigation is the correlation of mechanical properties and structural kinetic mechanisms (Hedworth and Stowell, 1971; Aran, 1979; Leterrier and G'Sell, 1967). In this paper however, a different approach that considers the relationship of stress to fiber adhesive energy with the matrix will be considered, in the determination of strain rate. The ratio of work of adhesion to the stress on the composite has been defined as the strain rate of the deformation of the composite (Smolej et al., 2009; Sinebe et al., 2019).

$$Strain\ rate = \frac{Work\ of\ Adhesion}{Tensile\ Strengt\ h} \quad (3)$$

Thus, to determine the strain rate, the work of adhesion (ΔF^{adh}) between the fiber (f) and the matrix (m) determined using the expression.

$$\Delta F^{adh} = \gamma_{mf} - \gamma_{mv} - \gamma_{fv} \quad (4)$$

Where: γ is the interfacial surface energy in mJ/m^2 and the tensile strength σ is in N/mm^2.

2.2 Materials and Method

Plantain fibers were extracted from plantain pseudostem by means of the manual scraper, prepared and treated with the following liquids: by Acetylation, Acetone, Glycerol, Hydrogen Peroxide, MEKP, Mercerization (NaOH), Methanol, Potassium Permanganate and Phosphoric Acid, to render the fiber surfaces hydrophobic. The matrix material was a polyester resin. The decision was made to mould cylindrical samples due to the greater potential for accuracy. The composite material was prepared in cylindrical forms.

2.2.1 Interfacial Free Energies

Contact angles were measured on the fibers and on the matrix materials using water and glycerol as probe liquid. Interfacial free energies were determined from contact angles by methods of Neumann, (1975); Fowkes, (1968); Sinebe et al., (2019) and the average values were obtained with which, in conjunction with equation 4, the work of adhesion was calculated for each treated material and presented in table 1. The tensile strengths of untreated and treated fibers were determined using testing machine D638 according to the ASTM guidelines and presented as shown in table 1.

3.0 Results and Discussions

It is noted from table 1 that the mercerization and MEKP treated samples have the maximum ultimate tensile strength, and at the same time have the highest free energies of adhesion. These results show that the adhesive bonding also leads to increase in tensile strength. Phosphoric Acid treated samples have the lowest tensile strength in agreement with the result that it has the lowest free energy of adhesion.

Table 1: Average values of calculated data

	Samples	ΔF^{adh} (mJ/m^2)	σ - UTS (N/mm^2)	Strain rate $\dot{\varepsilon}$ (10^{-9} s^{-1})	m Eq. (2)
	Unreinforced Polyester		148.18		
	Untreated	-40.3752	171.98	0.2348	0.2321
Treated	Acetone	-42.3604	190.41	0.2225	0.2362
	Acetylation	-44.1663	186.63	0.2367	0.2359
	Glycerol	-41.2407	177.07	0.2329	0.2334
	Hydrogen Peroxide	-42.3791	185.25	0.2288	0.2352
	MEKP	-45.8592	194.43	0.2359	0.2377
	Mercerization (NaOH)	-46.1778	198.49	0.2326	0.2385
	Methanol	-42.7380	179.29	0.2384	0.2342
	Potassium Permanganate	-42.0328	165.51	0.2540	0.2312
	Phosphoric Acid	-37.8042	148.84	0.2540	0.2264
				Average	0.2341±0.0035

Strain rate was calculated using equation 3 together with the relevant data and listed in table 1. From these results, using equation 2, the strain-rate sensitivity index was calculated and presented in table 1. It is seen to vary from 0.2264 for phosphoric acid treated fiber in the composite to 0.2385 for NaOH treatment fiber with an overall average value of m = 0.2341 ±0.0035. The strength of the bond between the matrix and the fiber was seen to be of paramount importance because that would determine whether the fiber would just pull out or remain in the matrix. The result of the analysis showed that the increase in work of adhesion led to an increase in the tensile energy, as expected and shown in figure 1.

Billmeyer (1984) suggested that, in regard to factors that influence the viscoelastic properties of plastics, that it is the magnitude of the cohesive energy density $U=\Delta E/V$ associated with a particular molecular structure that acts as the primary restraint on long chain flexibility. Because cohesive energy density is a function of intermolecular bonding forces, it is the strength of the dipole, dispersion and induction forces that most profoundly influence molecular mobility within a polymer. An understanding of the factors that determine the degree of steric hindrance and main chain flexibility is important to the successful selection and implementation of thermoplastics for specific design purposes in the industry.

The strain rate varies from $0.2225x10^{-9}$ s^{-1} for Acetone treatment to $0.2540x10^{-9}$ s^{-1} for potassium permanganate and phosphoric acid treatments. These values measure the rate at which the distances of adjacent parcels of the material change with time in the neighborhood of that fiber. Polymer materials exhibit this time dependent behavior in a load-bearing material. This is a viscoelastic phenomenon, which, in thermoplastics is of considerable importance to the design process in applications where the material is subjected to loading at a constant level of deformation for a prolonged period of time, such as filaments in tension and seals in compression (Gobble and Wolff, 1993). The low values of strain rates show that relative movements between the fiber and matrix, and also within the composite, are very low and that Potassium Permanganate and Phosphoric Acid treated systems are most probable to fail than the Acetone treated samples. The strain rate for the fiber reinforced polymer matrix was of the order of 10^{-9}/sec while that reported in the literature for polypropylene was of the order of 10^{-6}/sec. This result may suggest that the fiber sliding against the polymer matrix to which it was bonded was infinitesimally low and would not cause noticeable change in their bonding.

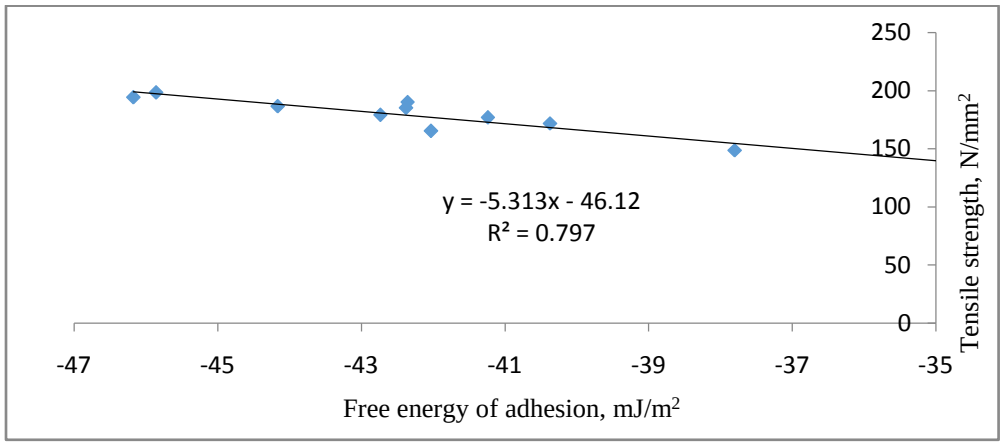

Figure 1: Effect of adhesive bonding on tensile strength of composite

The strain rate sensitivity index obtained in this work ranges from 0.2264 for Phosphorus Acid treated fiber to 0.2385 for NaOH treated fiber. For the untreated fiber, the sensitivity index is 0.2321. Since m values for Potassium Permanganate and Phosphoric Acid treated fibers are lower that the m values for untreated fiber, these two treatment liquids are the worst and should not be applied. NaOH and MEKP treated fiber have the lowest surface free energies of 31.24 and 31.55 mJ/m^2 showing that they are the most hydrophobic; they also have the highest m-values and therefore most desired for treatment of fibers for composite formation. Gobble and Wolff (1993) reported that m varied from 0.0260 to 0687for Polyvinyl Chloride (PVC) and from 0.1291 to 0.1316 for high density polyethylene (HDPE). Nwodo, et. al. (1988) studied the effects of interfacial free energy on creep of polypropylene in sodium dodecyl sulphate (SDS) surfactant solution and showed the strain rate to vary with interfacial free energy as given in figure 2.

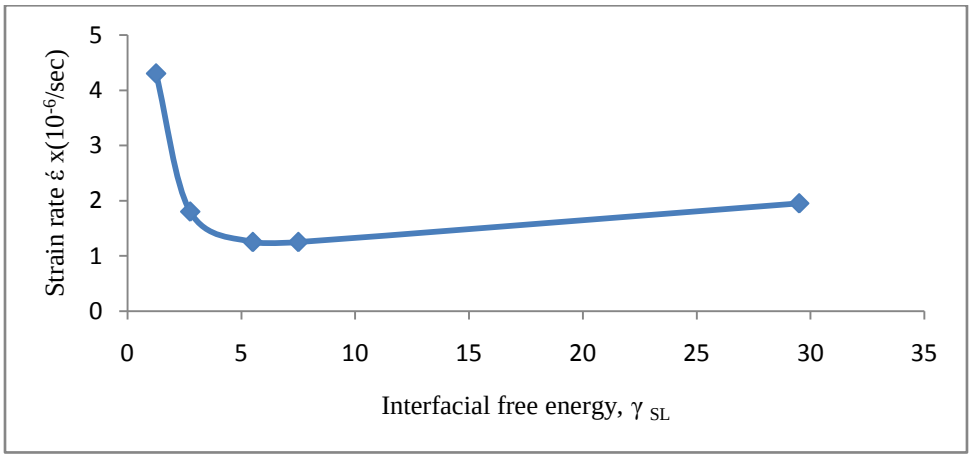

Figure 2: Relationship between strain rate and interfacial free energy for Polypropylene (Nwodo, et. al. 1988)

The behavior in figure 2 was attributed to the build-up of surfactant molecules at the surface of the polymer and the lowest point of the graph indicated the point of critical micelle formation (CMC). The CMC is an important characteristic of a surfactant. Before reaching the CMC, the surface free energy changes strongly with the concentration of the surfactant. After reaching the CMC, the surface free energy remains relatively constant or changes with a lower slope(Fuguet, 2005). This was demonstrated by Nwodo, et. al. (1988) as shown in figure 3.

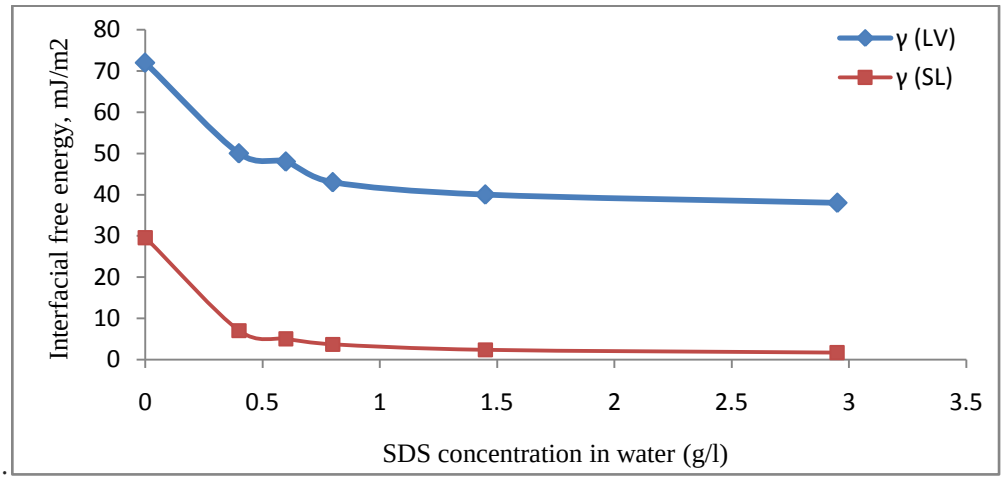

Figure 3: Interfacial free energy as a function of surfacxtant concentration (Nwodo, et. al. 1988)

Figure 3 shows that the CMC for this system occurs at about 0.5 g/l of surfactant in water at an interfacial free energy of about 5 mJ/m^2 corresponding to a strain rate of about 1.22 s^{-1} (figure 2). The tensile stresses corresponding to this point were obtained and used together with the corresponding strain rate value to calculate the strain rate sensitivity index,m, for polypropylene. It was found to vary from 0.1508 to 0.2186.

The values of m calculated in this work are somehow larger in many cases when compared with Nwodo et al, (1998) results. Taking the upper limits, the value reported in this work for the polyester composite is 10.44% higher than that calculated fromdata of Nwodoet.al. (1988) for polypropylene. The result of this work is also higher than that reported by Gobble and Wolff (1993) for high density polyethylene (HDPE) by44.8%. The differences could be attributable to variation in material type and surface treatment. Smolej, et. al. (2009), studying the strain-rate sensitivity and the activation energy of deformation in the superplastic aluminum alloy, showed that the m-values, determined on the basis of the true stress, true strain curves and also using the jump-test method, varied from 0.35 to 0.70, which depended upon the forming conditions. Majidi, et. al. (2017) also working with superplastic aluminum alloys demonstrated that the m-value is not constant and is highly dependent on the applied strain rate, strain level and testing method.

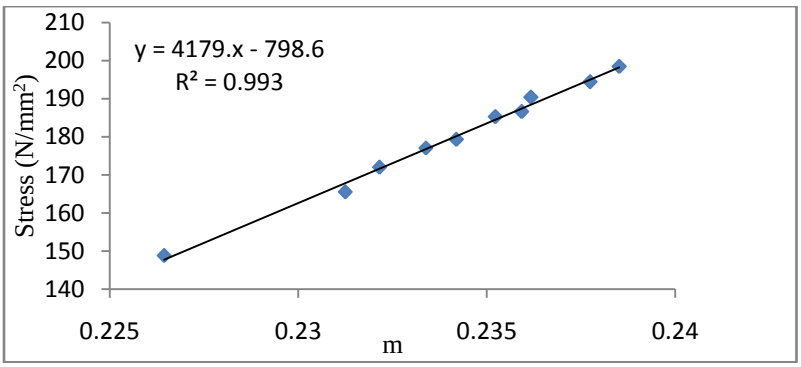

Figure 4: Relationship between stress and strain-rate sensitivity index

The relationship between the tensile strength and the sensitivity index, m, is shown in figure 4. That relationship, which is a linear one, is mathematically given in equation 5.

$$\sigma = 4179.7m - 798.67 \quad (5)$$

The value of $R^2 = 0.9935$ shows that the stress and the strain-rate sensitivity index are strongly correlated.

4.0. Conclusion

The strain rate was calculated from adhesive-tensile stress model and used to calculate the strain-rate sensitivity index. The low values of the strain rate suggest that the fiber sliding against the polymer matrix to which it was bonded was infinitesimally low and would not cause noticeable change in their bonding. Fiber-reinforced polymer composite structures for industrial applications would be more reliable if they show little or no deformation under load with time making the plantain fiber reinforced matrix a very good material for industrial applications. Since the strain rate sensitivity index, m, in this study is less than 0.30, it can be surmised that the reinforced plastic is relaxed and so would not be adversely subjected to stress build up at the interface between the fiber and the matrix. The strain-rate sensitivity index m is considered to be the most important parameter that characterizes superplastic deformation. For superplasticity, which is the ability of certain polycrystalline materials to achieve large elongations in a tensile test without necking prior to failure, large m-value is desired (Smolej, et. al. 2009), but for thermoplastics and viscoelastic materials, lower values of m suffice. This work also shows that the use of adhesive energy concept to determine the strain rate is valid since the values of strain rate indices calculated are comparable with values reported in the literature.

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