SRIRANGA B K
KIRTHAN L J
ARUN A MAGADUM
and
VISHWANATH PATIL

Investigation of mode II interlaminar fracture toughness of silicon-di-oxide filled glass fiber reinforced epoxy composites

Fracture is a new failure mode due to unstable propagation of a delamination crack growth in composite structures caused by applied load. The efficient characterization of the crack growth in composite structures enhances to estimate the duration of life. This paper mainly focusses on characterization of the fracture toughness of GFRP/epoxy matrix with SiO_2 filler form of composites and studying the crack behaviour in the (Mode II) ENF specimen which leads to interlaminar fracture in structural applications. The laminates are arranged to specify the Mode II interlaminar fracture toughness and interlaminar shear strength of GFRP/ epoxy matrix, GFRP/5% wt SiO_2, GFRP/10% wt SiO_2, using ASTM D7905 M-14 and JIS test methods.

The results show that unfilled SiO_2 GFRP/epoxy matrix composites are of more facture toughness; the result reveals that the JIS methods in plane shear Mode II test were lower value of fracture toughness than that of ASTM D7905 M-14 Mode II test. As recommend in ASTM standard, ENF (shear pre-cracked) specimen is applicable to measure Mode II fracture toughness.

Keywords: Interlaminar fracture toughness, shear pre-crack, mode II test method, mode II crack growth.

1.0 Introduction

The new class of engineering materials was emerged to match the gap between industrial and aerospace field of scope at various processing of material design. In this case, the natural fiber plays vital role in the polymer composites. The reason behind that they are renewable, biodegradable, cheaply available, and completely or partially recyclable. In the last few decades it is observed that research in failure and damage of composite samples due to impact loading have been documented and have significantly increased. During the impact loading of the experimental on composite structure material samples which leads to study on damage generation mechanism at basic conditions and it considered the main factors to decide the structural performance of the composites. Due to the nature of unbalanced distribution of stresses and anisotropic of composite material samples under the transient loading condition, which leads to damage processes of composite material samples [1]. Importantly considered are five main phases of failure mechanism under impact loading conditions [2-3]: (1) Due to high transverse shear stresses in top layer, which leads to propagate matrix cracking and fiber or matrix interface de-bonding damage mode; (2) Transverse bending crack in the layers of bottom, leading to strong flexure stresses; (3) Cracks propagation is restricted and diverted through the interlaminar region due to interlaminar delamination; (4) Under tension load leads to fiber failure damage mode and under compression load leads to fiber micro bucking and (5) Penetration of crack propagates.

Several experiments were carried out and studied on the measurement of resistance of delaminated composite material sample laminates and fracture toughness in different stages like, mode-I, mode-II and mixed of modes-I and II stress states. The ASTM D7905M-14 ENF standard test for opening mode-I [4], mixed modes I–II [5] and shearing mode-II [6] was determined. Russell et al. [7] have reported first about end-notched flexure (ENF) specimen was used by different modifications to measure fracture toughness in mode-II (G_{IIC}). The variation in specimen modification includes span, various forces, and truly existing segregation of delaminated faces to eliminate friction effects, propagated crack length, and calculation of G_{IIC}.

Specifications of specimen sizes and loading types are referred by the Japanese standards (JIS-K-7086-19937) [8] and by developed ASTM (D7905/D7905M-14) [9]. These methods in between specimen samples and studying loads are smooth, fascinating, and complicated frequently pre-owned.

Friedrich et al. [10] correlated to opening mode I and shearing mode II damage achievement of carbon fiber-epoxy

Messrs. Sriranga B K, Department of Mechanical Engineering, Don Bosco Institute of Technology, Kirthan L J, Associate Professor in Mechanical Engineering, RVCE, B'lore 74. Arun A Magadum, Associate Professor, Department of Mechanical Engineering, Don Bosco Institute of Technology, Mysore Road, Bangalore and Vishwanath Patil, Associate Professor in Mechanical Engineering, Sharanbasappa University, Kalburgi., India. E-mail: srirangabk@gmail.com / contactkirthan@gmail.com / arunmagadum123@gmail.com / vishwanath.patil09@gmail.com

and polymer thermoplastic based on polyether ether ketone. It was displayed that critical strain energy of mode I, G_{IC} and mode II, G_{IIC} were ten times more in the case of carbon fiber/ polyether ether ketone compared to carbon fiber/matrix epoxy composite laminate.

Hunston et al. [11] carrying a detailed examined between polymer thermoplastic composites and strengthen-polymer thermoset, demonstrate that strengthen resin matrices composites are increased in a delamination of interlaminar fracture strength by 1 kJ/m^2 correlated to the accurate epoxy resin composite laminates.

Floros [12] characterized the opening mode I, shearing mode II and mixed mode (I+II) critical stain energy release rate on composite bonded joints experimentally and numerically. It showed that joints with film adherent have much larger delamination of fracture toughness than adhesive joints which exhibited brittle behaviour.

Fernandez [13] fatigue crack/fracture toughness characterization for mixed mode I+II of carbon fibers/epoxy composite adhesive bonded joints are achieved by making use of the single point End Notch Flexural bending test.

Paris law was used to establish relation between fatigue crack growth rate and the fluctuation of the critical strain energy release rate are investigated. Persistent results were obtained for lower force level.

The sequence of initiated and generation criteria yield a traditional estimation of the load transmit capability of a structural component. In addition, the development toughness, durability and perceptivity of the results with recognition to a variation in the interface properties, and the nonlinear structural failure effected by the inter delamination crack growth process are computed.

This research paper investigates the fracture mechanics criterion standard test methods to characterize the fracture resistance as a generic property of a material.

Investigate highlights shearing mode II (ENF) interlaminar fracture of a proprietary fabric glass/epoxy with silicon dioxide composite material system.

2.0 Experimental work

2.1 Materials used

The present study materials used in this paper are base matrix phase of epoxy resin as LY556 and HY591 hardener were purchased from Chemist Engineers Limited, Bangalore, India. For reinforced phase as bidirectional thickness 0.4mm was used and S-glass fiber in woven as 280gsm supplied by Suntech Fiber Private Limited. Silicon dioxide (SiO_2) powder manufactured and supplied by Loba Chemie Pvt. Ltd., Mumbai, India was used as filler material in the present study. Silica (silicon dioxide) occurs commonly in nature as silica sand or quartzite, sandstone. It is starting material for the production of silicate glasses and ceramics. The chemical composition and physical properties of epoxy/S-glass fiber/ silica fillers as shown in tables [11].

Table 1: The chemical composition of S-glass fiber

Elements	SiO_2	Al_2O_3	MgO
Percentage (%)	65	25	10

Table 2: Thermo-physical parameters of S-glass fiber

Physical properties	Specifications
Thermal expansion (mm/m ^oC)	5.26
Density of S-glass fiber (kg/m^3)	2.49
Young modulus (GPa)	89
Elongation at break (%)	5.4
Tensile strength (MPa)	4750

Table 3: The chemical composition of epoxy resin at 23^{o}C

Elements	Resin	Hardener	Aluminum	HNT
Percentage (%)	63	27	10	-

Table 4: Thermo-physical parameters of silica filler

Physical properties	Specifications
Particle size (mesh)	40-150
Density of S-glass fiber (kg/m^3)	2.20
Molecular weight	60.08
Physical state	Crystalline powder
Colour/Odour	White

2.2 The process of vacuum bag molding techniques

The composite plates were studying buildup from 08 layers of fabricate the plain glass fabric using polyester epoxy resin and silicon dioxide fillers are added. The three plates of composite laminates were manufactured jointly from 0 wt% of SiO_2, 5 wt% of SiO_2, 10 wt% of SiO_2 filler particles and polyester epoxy resin. Beginning, 08 plies of interwoven glass fibers reinforced with polyester epoxy resin of laminate plates was manufacture by using hand layup assisted vacuum bag molding process, hereby a fiber glass plate mold was heated by a autoclave under temperature restrained heating pad placed. Previously sealing the vacuum bag, PTFE (Teflon film) 12.5μm deep (0.50 mm thickness) was also added into middle layers (after 4 layers of glass fabric) at corner boundary of the laminate to maintain a pioneer pre-crack gap. The stacking of glass fibers reinforcement was controlled against the mold with two vacuum bags i.e. inside bag and outside bag airtight to the mold and the polyester resin was implant into the reinforcement stacked using atmospheric pressure inner vacuum bag is 7.5 milli bar, where outer the bag atmospheric pressure is around 1.5 bar. Lower surface of the fiber glass reinforced plate mold was supported at 50ºC over resin mixture.

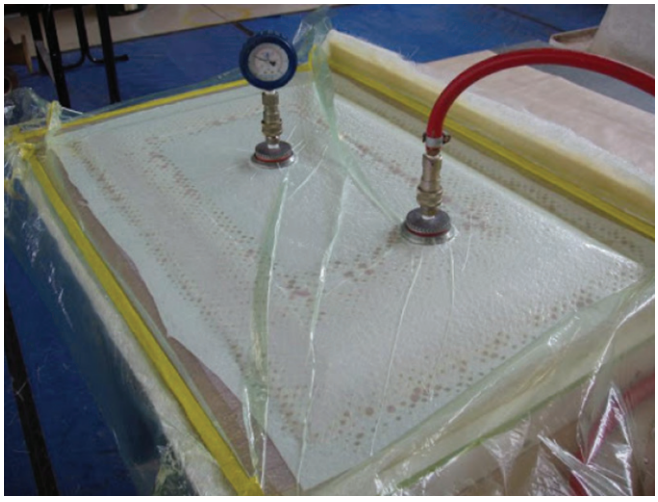

Fig.1: Vacuum bag molding process setup

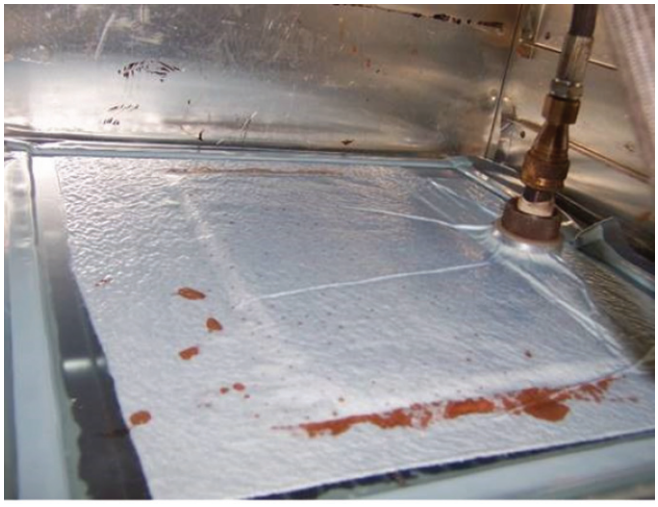

Fig.2: Preparing the specimen by using vacuum bag molding process

Fig.2a: Sample 1 - Glass fabric reinforced with polyester resin (GF+0% SiO_2)

Fig.2b: Sample 2 - Glass fabric reinforced with polyester resin (GF+5% SiO_2)

The specimens are prepared by vacuum bag process and also take 24 hrs for healing after the complete infusion of fiber reinforcement resins. Finished GFRP with silicon-di-oxide laminates in the average of near 60% fiber volume fraction of thickness specimen is 3.0mm.

3.0 Delamination of opening mode II fracture toughness test (ENF test)

The laminates of hybrid composites stacking sequencing are established by varying 5%, 10% of silicon-di-oxide with orientation of 0/90°. In these composites, the epoxy resin keeping balancing with E-glass fiber to study the interlaminar delaminations fracture characterization.

Fracture toughness testing was conducted by the three methods: standard end notch flexural, Japanese industrial standard and ASTM D7905M-14. The ASTM D7905M-14 test consisted of non-pre-crack and pre-crack fracture toughness tests and determines compliance calibration equation for both non-pre-crack and pre-crack were implemented the each test specimens.

The compliance calibration tests were operated to higher loads of about 25% of critical load so that the span did not begin to grow. Compliance calibration evaluation and calculation of constants (A and m) of the compliance equation. All tested specimens are denoted with single end notch bending (SENB) carried out in a zwick roell test machine using end-notched flexural (ENF) specimens with 165 mm length and 25 mm width, as shown in Fig.3.

The compliance calibration (CC) of SENB specimens are marked were built from the corner tip of the (PTFE) Teflon sheet film inserted (a=20 mm, a=30 mm and a=40 mm), as shown in Fig.3. The tested specimens have a beginning 50

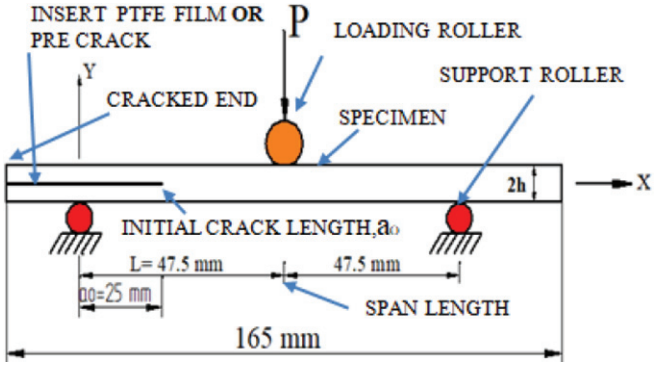

Fig.3: Specimen dimension

TABLE 5: THE POLYMER LAMINATES STACKING SEQUENCE

Composites	Composition
GE	Glass fiber + Epoxy resin
GE+ 5% SiO_2	E-glass fiber (55%) + Epoxy resin (40%) + SiO_2 (5%)
GE + 10 SiO_2	E-glass fiber (50%) + Epoxy resin (40%) + SiO_2 (10%)

mm deep mid plane pre-crack at one end edge to pre-initiate the span.

These ENF standard tests specimens are composed of non-pre-cracked (NPC) and pre-cracked (PC) fracture tests and main objective to determine the $G_{IIC–}$ non-pre-crack and $G_{IIC–}$ pre-crack from SENB specimen by using compliance equation for both non-pre-crack and pre-crack test specimens. The initial non-pre-crack test was implementing for the a_o=20 mm and also the span length between the holdup is 95 mm for test specimens. These testing specimens were arranged on the three point single end notch bending test device and the notch was sustained in the center of the left side of the supported device. When the force P arrive a pre-determined values, which does not pre-initiate the delamination, it required that test must be completed and also the test specimen was repositioned and withdrawn at different a_o position in device. The very same method was embraced for the another position at a_o=30 mm and last third test position marked at a_o=40 mm. At final stage, the implementation is until the ENF test specimen and was created inter delamination. The test specimen is interrupting, when the load, P value extended the highest value. After finishing the non-pre-crack fracture test, which generates an opening shear pre-crack, the test specimen was taken out from the testing machine and repeated to conduct pre-crack test with the crack length measures. The new delamination crack tip position was marked and pre-crack fracture tests were determined using new snap length with the same measures for the non-pre-crack test, except the loads and displacement data were dissimilar. The compliance calibration equation (Equation [1]) was established by experiments to forecast crack length as required. The calibration coefficient (CC) of A and m are resolute from the straight regression data of the constant CC curve as function obtained by Equation (1).

$$C = A + ma^3 \quad \text{... (1)}$$

Where, (C) is the compliance, (a^3) is cubed crack length and (A) is the intercept and (m) the slope of the linear regression.

From these results data, using the constant m from the calibration compliance equation, the values of displacement and forces deliberate to obtain the shear pre-crack of mode II, G_{IIC} according to equation (2).

$$G_{IIC} = 3m \frac{(Pc\ a)^2}{2\ B} \quad \text{... (2)}$$

Where, (P_{max}) the maximum forces that beginning the cracking/delamination, (a_o) the crack length to initial delamination (25 mm) and B as width of specimen.

4.0 Mode II fracture toughness test calculations

4.1 According to JIS standard

$$G_{IIC} = 9 \frac{(F\,a)^2}{2B} \frac{C}{(2L^3+3a^3)} \quad \text{... (3)}$$

$$\frac{9\,[475*27.315]^2}{2*25} \left[\frac{0.021053}{2(40)^3+3(27.315)} \right]$$

G_{IIC} = 4216.06 J/m^2.

where,

G_{IIC} = Critical strain energy release rate for mode II in J/mm.

a = Crack length in mm

B = Specimen width in mm

L = One half span of specimen in mm

P_C = Critical load of failure in Newton (N)

4.2. According to ASTM D 7905 standard

$$G_{II} = \frac{9\,F\,\delta\,a^2}{2B\left(\frac{1}{4}L^3+3a^3\right)} \quad \text{... (4)}$$

$$G_{IIC} = \frac{9*475*13*(27.315)^2}{2*25 \left[\frac{1}{4}(40)^3 + 3(27.315)^3\right]}$$

G_{IIC} = 1695.495 J/ m^2.

G_{IIC} = 1695.495 J/m^2.

5.0 Results and discussions

5.1 Fracture toughness of ENF test

In order to apprehend the variations inside the mechanical properties between silicon dioxide and epoxy/GF composites,

Table 6: Specimen number and geometric parameters.

Tests	Specimen	a_o (mm)	2 h (mm)	B (mm)	S (mm)	L_T (mm)	L_i (mm)
JIS	GF+0% SiO_2	25.0	3.00	25.00	95.00	165.00	50.0
	GF+5% SiO_2	25.0	3.00	24.50	95.00	165.00	50.0
	GF+10% SiO_2	25.0	3.00	24.70	95.00	165.00	50.0
ASTM D7905M-14 NPC	GF+0% SiO_2	25.0	3.00	25.00	95.00	165.00	50.0
	GF+5% SiO_2	25.0	3.00	24.50	95.00	165.00	50.0
	GF+10% SiO_2	25.0	3.00	24.70	95.00	165.00	50.0
ASTM D7905M-14 PC	GF+0% SiO_2	25.0	3.00	25.00	95.00	165.00	50.0
	GF+5% SiO_2	25.0	3.00	24.50	95.00	165.00	50.0
	GF+10% SiO_2	25.0	3.00	24.70	95.00	165.00	50.0

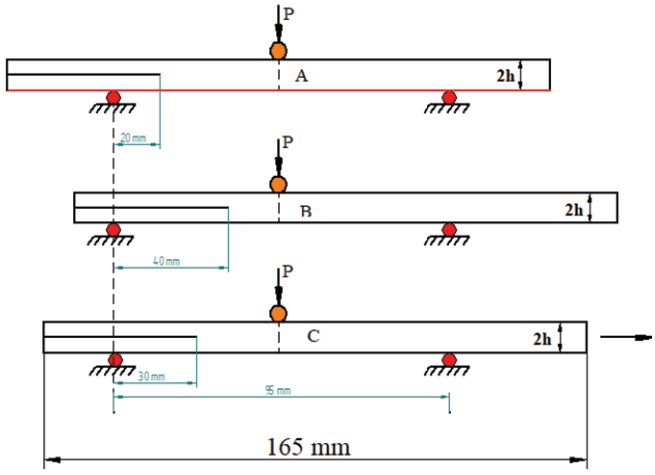

Fig.4: Specimens with compliance calibration marks at a_O = 40 mm, a_O = 30 mm and a_O = 20 mm.

mode II interlaminar fracture toughness become investigated. Tables 7 and 8 sum up the determined estimations of the compliance calibration CC for non-pre-crack and pre-crack tests, and the CC coefficients m and A. These coefficients were resolved utilizing a linear least squares straight regression evaluation of the compliance, C, versus break length cubed, a^3, records from equation (1). Figs.5 and 6 display a linear suit to the average data points of non-pre-crack and pre-crack tests for silicon dioxide and polyester resins, respectively. From these outcomes, and according to equation (2) become feasible to gain the G_{IIC} values.

5.1.1. Load vs displsacement curve

The testing specimen shows that load versus displacement curve for the composition of two samples are indicated in Fig.7. The values and figures are presented in Tables 7 and 8, from these data, initial response from peak load for both pre-crack and non-pre-crack tests for the epoxy glass fibers laminates are persistently high. In the beginning, the curve is linear at non-pre-crack test for both laminates, so it is indicating elastic loading. Thereafter, test specimens are linear growth of the curve in pre-crack test, position is an instantly keen load let fall, it shows that the unstable crack propagation occurs. Fig.7 shows load-displacement curves in between glass fiber and filler particles of silicon di-oxide, make the nano silicon di-oxide fillers in the mid plane of ENF specimen among 8 layers has main significant effect on fracture surface of the inter delamination prior to crack propagation. Hence, linear curves of all test specimens are near to the same, in fact, the displacement of all test specimen are dissimilar due to adding percentage of small fillers. The crack growth effortlessly propagated in GFRP laminates. The rate of stored strain energy release rate is adequate to crack propagate at the mid plane of test specimen. Whereas, crack propagates within the GFRP composite laminates by minor incremental bound with decreasing the loads. So that, the glass fiber reinforcement of polyester resins laminates are more dominant across nonlinear region due to fibers bridge. It shows Tables 7 and 8 represent the ENF test specimen of mode II delamination of fracture toughness values for them composite laminates and their compliance coefficient of variations based on the values from Table 7. In the non-pre-crack test specimens are interrupted before the delamination propagation, it presented matrices different changes in the G_{IIC} values. As per Table 8 and Fig.7 it is observed that the average G_{IIC} values for the silicon dioxide/carbon fiber composites in the non-pre-crack test were lower than the values found for pre-crack test composites.

Initially when the specimen is origin, i.e. in a unload condition where the position is at origin the load withstanding capacity of the specimen is 0.0751N.

As on changing the position of loading point from 0mm to 5mm, the specimen started to elongate and showed a cracking path by withstanding a load of 304N.

Further on changing the position of loading point from 0mm to 10mm, the specimen crack further propagates

TABLE 7: JIS FRACTURE TEST

Test	Specimen #	Measured a_i (mm)	B (mm)	P_c (N)	d_c (mm)	C (mm/N)	G_{IIc} (J/m^2)
JIS	GF+0% SiO_2	27.31	25.00	475	2.74	0.019464	4216.06
	GF+5% SiO_2	31.21	24.50	360	2.68	0.023485	3815.89
	GF+10% SiO_2	30.47	24.70	329	2.70	0.023464	3035.61

TABLE 8: ASTM D7905M-14 NPC &PC FRACTURE TEST RESULTS

		Measured					
Test	Specimen #	a_i (mm)	B (mm)	m	P_c (N)	d_c (mm)	G_{IIc} (J/m^2)
PC	GF+0% SiO_2	27.31	25.00	1.53E-06	475	2.74	1618.15
	GF+5% SiO_2	31.21	24.50	1.98E-06	360	2.68	1202.85
	GF+10% SiO_2	30.47	24.70	2.15E-06	329	2.70	1090.86
NPC	GF+0% SiO_2	30.47	25.00	1.53E-06	851.5	2.35	844.0
	GF+5% SiO_2	30.47	24.50	1.98E-06	890.8	2.25	849.9
	GF+10% SiO_2	30.47	24.70	2.15E-06	907.8	2.34	895.1

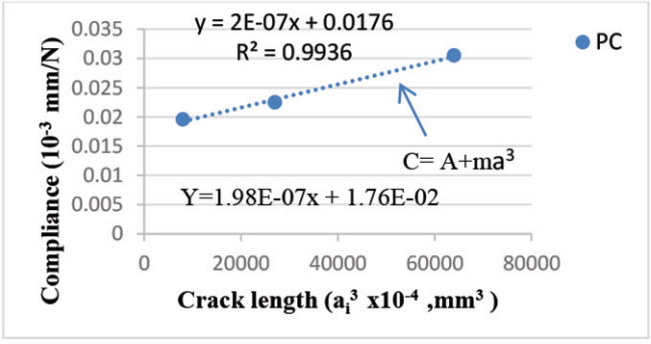

Fig.5: Compliance Cc vs. crack length cubed for polyester resins glass fabric laminates in pre-crack tests

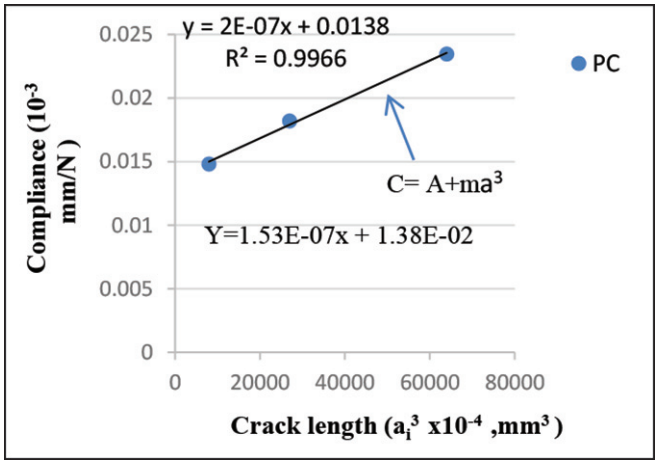

Fig.6: Compliance vs. crack length cubed for silicon dioxide fillers/ polyester resins glass fabric laminates in Pre-crack tests

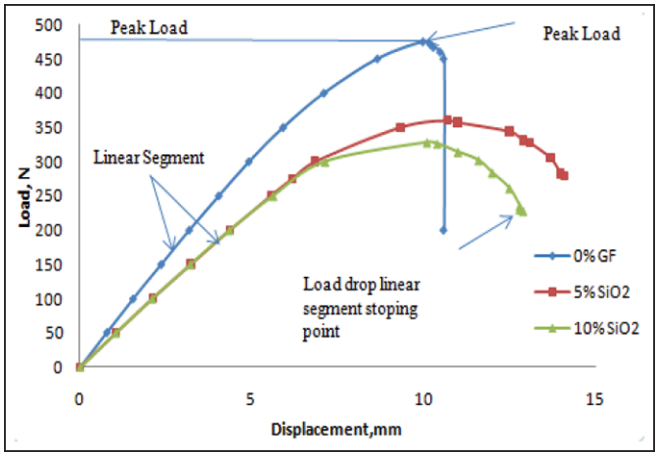

Fig.7: Load-displacement curves for pre-cracked (PC) and non precracked (NPC) tests for the GF/SiO_2 composites

constantly where at this point the specimen was at this ultimate strength of load withstanding capacity P max = 475N with a maximum elongation of 10mm.

Finally the specimen reaches critical loading conditions where it started to deform/damage to its capacity.

As we all know the critical loading conditions is nothing but the quantitative estimate of an exposure to one or more loading capacities below which significant harmful effects on specified sensitive elements of the material would deform.

On observing the specimens behaviours at different loading conditions of the loads were gradually increasing the specimen showed the elongation correspondingly, i.e. graph plotted as shown in Fig.8. From the above graph one can analyze that the composite material of study shows that it is directly proportional to the load with corresponding elongation. But as we followed literature survey of Dr. Kunigal Shivakumar [10] it confirmed us that the composite material may show different characteristics at varying load conditions also it may vary as per the standards maintained at the loading conditions. In order to meet this convenience we may introduce a constant CC which is referred as compliance in ASTM standards and JIS standards. This compliance introduced may satisfy the physical law of load is directly proportional to the elongation. In order to satisfy these conditions first we have to calculate compliance. i.e. in the material testing of our specimen we got the compliance value C is 0.0210526mm/N. we got A/a = C 1 /Co = 0.0210526mm/N. On calculating the compliance we reach the conditions of physical law. i.e., L $\alpha\delta$.

6.0 Conclusion

The woven fabric of E-glass fiber and epoxy composite of silicon dioxide is prepared with three different wt. % of silicon dioxide via 0% wt., 5% wt. and 10% wt. from these paper, following observations were made:

1. The crack voids particularly determine the fracture toughness and also the performance of the composites laminates at the working area. The high void contented mean less fatigue resistance, greater sensitivity to water penetration. Even so existence of void is inevitable in vacuum bag process. In the current examination it was observed that the inclusion of silicon dioxide into woven fabric epoxy composites shows more voids contents compared to unfilled composites.
2. It is observed from the ENF tests that interlaminar fracture toughness for GF+0% SiO_2 showing 4216.06 J/m^2 and decreases with the additions of 5 and 10 wt. % of silicon dioxide filled glass fabric epoxy composites. High void contents and non uniform dispersion of silicon dioxide through the laminate may be the reason for decreases in interlaminar fracture toughness.
3. The existence of silicon dioxide filler provides crack point blunting by shear deformation process nearby the crack tip. Hence, it can be concluded that by adding of silicon dioxide fillers there is decrease in delaminations fracture toughness of glass fabric reinforced epoxy matrix compared to 0 wt%.

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