Fracture Toughness of Particle Filled Fiber Reinforced Polyester Composites

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SYNOPSIS

Fracture behavior of polyester composite systems, polyester mortar and glass fiber reinforced polyester mortar, was investigated in mode I fracture using single edge notched beams with varying notch depth. The beams were loaded in four-point bending. Influence of polymer content on the flexural and fracture behavior of polyester composites at room temperature was studied using a uniform Ottawa 20–30 sand. The polymer content was varied between 10 and 18% of the total weight of the composite. The flexural strength of the polyester mortar systems increase with increase in polymer content while the flexural modulus goes through a maximum. The critical stress intensity factor (K_{IC}) for the optimum polyester mortar (14%) was determined by two methods including a method based on crack mouth opening displacement. The K_{IC} for polyester mortar is linearly related to the flexural strength. Polyester mortar (18%) reinforced with 4% glass fibers was also investigated, and crack growth resistance curve (K_R) was developed with crack extension (Δa). A model has been proposed to represent the fracture toughness with change in crack length, $K_R - \Delta a$ relationship, of fiber reinforced polyester composite.

INTRODUCTION

Particulate filled polyester composite such as polyester mortar is formed by combining mineral aggregates such as sand or gravel with a polymerizing monomer. In recent years polymer mortar systems are being used increasingly in the construction and repair of structures, highway pavements, bridge decks and waste water pipes.[1] The versatility in formulating and processing with rapid setting and high strength properties has resulted in the use of polyester mortar in diversified applications. The current use of polyester composite warrants mainly fine aggregates that are no more than 5 mm.[1] As a structural and repair material polyester composite, which is invariably brittle, must withstand high stresses under extreme service conditions. Hence a knowledge of the fracture properties is important in aiding the efficient utilization of polyester composites.

Linear elastic fracture mechanics (LEFM) parameters have been used in characterizing the fracture properties of polymers, cement mortars, cement paste and cement concrete.[2-7] The application of LEFM to cementitious materials was justified by treating crack branching and fracture process zone to be analogous to a small size plastic zone at the crack tip. However satisfying the ASTM E 399 requirements for plane strain fracture toughness testing (K_{IC}) in cementitious materials involves fabrication of very large specimens that are not easily amenable for laboratory testing. Despite these limitations, LEFM is still being used extensively in determining the critical stress intensity factor based on measurements of the initial notch depth and load to failure.[4] Direct measurement of crack extension is seldom possible in mortar or concrete systems without recourse to expensive instrumentation. Crack extensions are determined indirectly from calibration curves of load–displacement (P–δ) or load crack mouth opening displacement (P–CMOD) responses.[8,9]

Fracture studies on concrete-polymer composite materials have been primarily limited to polymer-

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Table I Composition of Polyester Composites

Polymeric Matrix	By Weight
Polyester	
Polyester Dion Iso-6315 (Koppers Co., PA)	10–18%
Methyl ethyl ketone peroxide (initiator)[a]	2%
Cobalt napthenate (promoter)[a]	0.2%
Aggregate	
Ottawa 20-30 Sand	82–90%
Fibers	
Glass Strands (13 mm long)	0–4%

[a] By weight of resin.

portland cement concrete (PPCC) and polymer impregnated concrete (PIC).[10-12] Cook and Crookham[10] observed that K_{IC} and stiffness decreased in PPCC, regardless of the polymer type, mix and treatment. Polymer impregnation on the other hand increased the fracture toughness and failure strain of the concrete. Polymer impregnated concrete was found to be notch sensitive with K_{IC} having a limiting value between notch-to-depth ratio of 0.35 and 0.42 and beyond which K_{IC} generally decreased. Alezksa and Beaumont[11] have observed that the work to fracture was enhanced in the concrete systems when impregnated with an acrylic polymer. In an earlier study Tazawa and Kobayashi[12] noted that the improvements in PIC properties could be reasonably described using the Griffith theory. Results on the fracture behavior of epoxy mortar have been recently reported by Vipulanandan and Dharmarajan.[13,14] While the fracture parameters of epoxy mortar increases with increase in polymer content, the notch sensitivity is reduced. The K_{IC} of epoxy mortar also reduces with increase in temperature.[14,15]

The validity of K_{IC} as a fracture criteria for polyester mortar can only be established if it can be shown that tests on specimens with significantly different dimensions (notch depth, specimen size) yield identical results within experimental errors. In order to characterize the mode I fracture behavior of polyester composite, single edge notched beams (SENB) were used in this study. Methods based on crack mouth opening displacement (CMOD) are used in determining the critical stress intensity factor (K_{IC}) for the polyester mortar formulations with varying resin contents. The fracture toughness of polyester mortar is related to the flexural strength. Resistance curve (K_R) for 4% glass fiber reinforced polyester composite is also developed.

EXPERIMENTAL

The composition of polyester composite systems are summarized in Table I. The Ottawa 20–30 sand (ASTM C-190) was composed of quartz with the particle size varying between 0.59 and 0.84 mm. The coefficient of uniformity (ratio of D_{60}/D_{10}, where is D_{60} and D_{10} are the aggregate sizes that corresponds to 60 and 10% of sample passing by weight), which is a measure of particle gradation, was 1.08. The room temperature viscosity of the polyester monomer varied between 40 and 50 P. Polyester composite specimens were compacted in three layers in a teflon lined aluminum mold of dimensions 300 × 50 × 50 mm. All the fracture studies were performed on 33 mm thick specimens. The specimens were first cured at room temperature for a day and at 60°C for an additional day prior to testing. The samples were notched using a diamond saw (2 mm thick) to a maximum depth of 38 mm. During the test the cross head speed of the closed loop servo hydraulic testing machine was maintained constant at 0.05 mm/min. The composite specimens were tested in four-point bending (third-point loading) as shown in Figure 1. The deflection at the center of the beam was measured at the top of the specimen using an LVDT accurate to 0.002 mm. The crack mouth opening displacement (CMOD) was measured using a COD gage clipped to the bottom of the beam and held in position by two aluminum knife edges glued to the specimen (Fig. 1). For every test, both the load versus load-point deflection and the load versus crack mouth opening displacement were monitored continuously using X–Y recorders.

Unfilled polyester resin beam specimens (25 × 25 × 200 mm) were also tested in four-point bending to determine their mechanical properties. At room

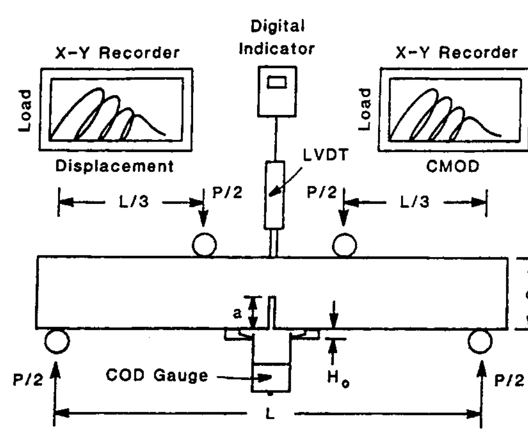

Figure 1 The four-point bending test configuration.

temperature the unfilled polyester polymer had a mean flexural strength of 81 MPa with a standard deviation of 6 MPa.

BEHAVIOR OF POLYESTER COMPOSITES

The variations in flexural strength and modulus of polyester mortar systems are shown in Figure 2. At room temperature the flexural strength of polyester mortar increases at a decreasing rate with increasing in polymer content as shown in Figure 2(a). The flexural modulus increases with increasing polymer content; but on reaching a maximum it decreases with further increase in polymer content [Fig. 2(b)]. Within the range of polymer contents investigated the 14 and 18% polyester mortar systems can be characterized as highest modulus and highest strength respectively.

Notch Sensitivity

A material may be considered notch sensitive if the presence of a notch causes a change in the strength

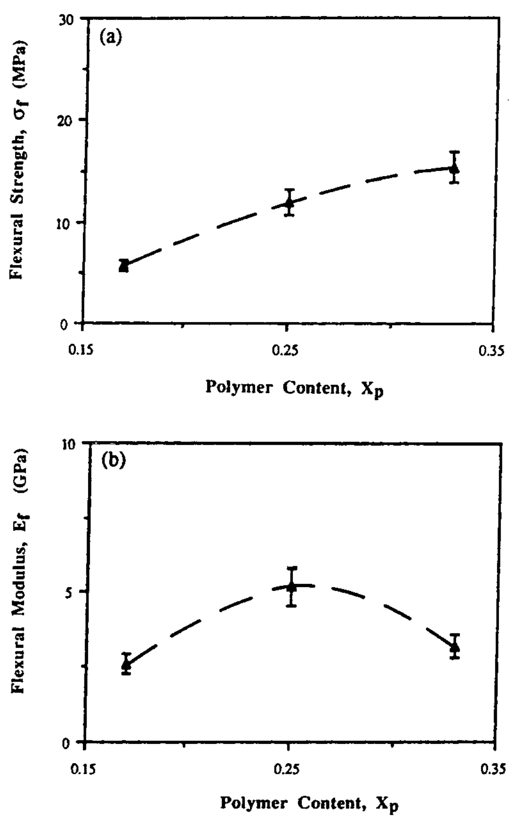

Figure 2 Variation of flexural strength and modulus of polyester mortars.

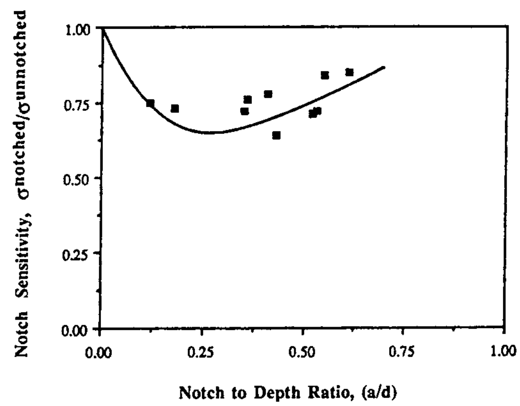

Figure 3 Notch sensitivity of 14% polyester mortar.

of the material (calculated on the reduced cross section, but neglecting the stress concentration effect of the notch). Typical flexural load–displacement relationships for polyester mortar with and without notch have been reported by Vipulanandan and Dharmarajan.[16] For cementitious composites notch sensitivity refers only to the possible reduction in strength due to the presence of the notch. Figure 3 shows the variation in notch sensitivity of 14% polyester mortar (ratio of notch strength to unnotched strength) with relative notch depth. The 14% polyester mortar when notched showed maximum reduction in strength of about 25% of the unnotched strength. Similar trends have also been observed in cement paste, cement concrete and epoxy mortar.[4,13,14]

Determination of K_{IC}

There are several methods available for determining the critical stress intensity factor K_{IC} based on initial notch depth and effective crack length. As nonlinearity is observed near the peak load it is of interest to determine both the crack extension and the crack length at the peak load. In this regard, an indirect estimation of the crack extension based on CMOD measurements[8,9] will be used to determine the critical stress intensity factor of polyester mortar. Stress intensity factors obtained from effective crack length will be compared to the results obtained using initial notch depth method.

Initial Notch Depth Method

According to the ASTM E 399 recommendation, the critical load for evaluating a valid K_I value is determined from the load displacement plots by the secant line with a slope of 0.95 times the initial tangent slope. Instead of this procedure, the maximum load is utilized in determining K_I and this would result in a slightly higher K_{IC} value. A similar approach has been used in determining the stress intensity factor of cementitious materials.[4] Assuming a beam of cross section $b \times d$ with an initial crack length a, the stress intensity factor was calculated by using the equation for four-point bending developed by Brown and Srawley.[17] The relationship is as follows:

$$K_I = 6Ma^{1/2}Y(a/d)/bd^2 \qquad (2a)$$

where

$$Y(a/d) = 1.99 - 2.47(a/d) + 12.97(a/d)^2 - 23.17(a/d)^3 + 24.80(a/d)^4 \qquad (2b)$$

and M is the applied pure bending moment. Figure 4(a) shows the variation of K_I polyester mortar with initial notch depth and the response is approximated by a horizontal line. The critical stress intensity factor (K_{IC}) is the limiting value of K_I. In order to satisfy the ASTM E 399 recommendation (plane strain fracture toughness of metallic materials) for specimen thickness, b ($>2.5(K_{IC}/\sigma_y)^2$), and hence the polyester mortar should have a minimum thickness of 18 mm (σ_y is the flexural strength of polyester mortar). This condition was satisfied by adopting 33 mm thick specimens in this study. The ASTM E 399 also has a recommendation for crack length a ($>2.5(K_{IC}/\sigma_y)^2$). This criterion will result in plain strain crack lengths of 18 mm for polyester mortar. The test results (Fig. 4) indicate that the plain strain crack length requirement of ASTM E 399 is also satisfied by the polyester mortar. The variation of the K_{IC} with mortar strength for the polyester is shown in Figure 5. The fracture toughness of polyester mortar increases linearly with increase in mortar strength.

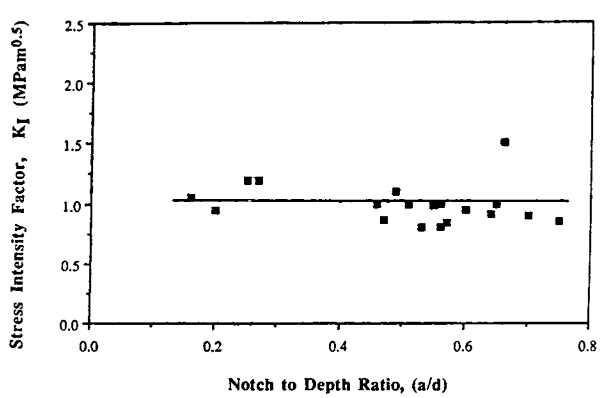

Figure 4 Variation of stress intensity factor of 14% polyester mortar with initial notch depth.

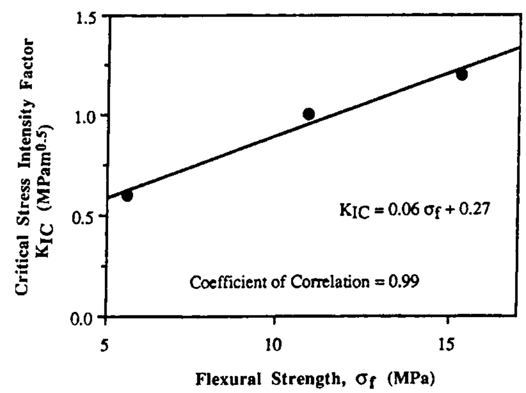

Figure 5 Variation of critical stress intensity factor with flexural strength of the polyester mortars.

CMOD Method

Using LEFM, the relationship between elastic crack mouth opening displacement ($CMOD^e$) and the corresponding crack length in four-point bending can be represented as follows[18]:

$$CMOD^e = 4\sigma a V(\alpha)/E' \qquad (3)$$

where s is the net stress ($6M/bd2$) with M being the applied pure bending moment, α equal to (a + Ho)/(d + Ho) with Ho being the clip gage holder thickness and E' is equal to E (modulus) for plane stress and $E/(1 - \nu^2)^{0.5}$ for plane strain and ν is the Poisson's ratio. An empirical formula with 1% accuracy for any a is used to calculate $V(\alpha)$[18] and is expressed as

$$V(\alpha) = 0.8 - 1.7(\alpha) + 2.4(\alpha)^2 + 0.66/(1 - \alpha)^2 \qquad (4)$$

Hence if during slow crack growth $CMOD^e$ could be determined at various loading levels it would be possible to determine the corresponding crack length. A schematic plot of load (P) versus crack mouth opening displacement (CMOD) is shown in Figure 6. If the material behaves elastically up to peak load without any crack extension, the relationship between load and CMOD will be linear. However by unloading the specimen just prior to the peak load and immediately after the peak load as shown in Figure 6, it can be seen that there is inelastic displacement associated with the response of the notched beam. At peak load, the total CMOD is composed of the elastic displacement (no crack ex-

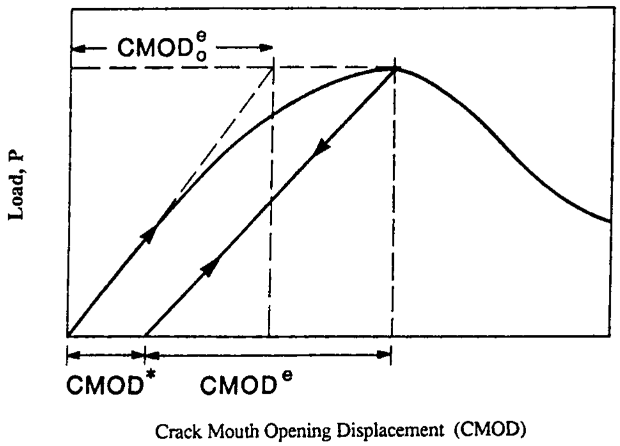

Figure 6 Schematic of load–CMOD response showing loading and unloading characteristics.

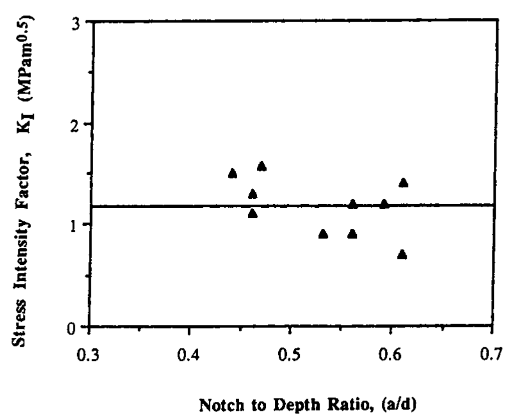

Figure 7 Variation of effective critical stress intensity factor of 14% polyester mortar with initial notch depth.

tension), inelastic displacement and the elastic displacement due to slow crack growth. The nonlinear displacement observed in the P–CMOD response can be attributed to both creep and slow crack growth. The inelastic displacement that occurs during crack growth in particulate composites with very high percentage of aggregate fillers is possibly due to friction associated with roughness of cracks and aggregate interlocking. In order to apply LEFM the inelastic CMOD should be extracted from the total CMOD at peak load. The elastic CMOD^e at peak load could be obtained by unloading the specimen at 95% of the peak load.[8,9,15]

Using the initial compliance (C_i = CMOD/P) and the unloading compliance (C_u, measured at about 95% of peak load) in eq. (3) and by combining the resulting equations, the effective crack length (a_e) is determined using the following relationship:

$$a_e = a_i(C_u/C_i)[V(a_i)/V(a_e)] \qquad (5)$$

A numerical iterative procedure was used to determine ae from eq. (5). The effective crack length was then used in eq. (2a) to determine effective K_I. The variation of effective K_I with the initial notch depth is shown in Figure 7. The results based on initial notch depth and effective crack length are summarized in Table II. The crack extension ($\Delta a = a_e - a_i$) varied between 5 and 15% of the initial notch size. There is an increase in the mean value of K_{IC} owing to the correction in crack length.

CRACK GROWTH RESISTANCE CURVE

Glass fiber reinforced polyester composite shows substantial amount of nonlinearity and stable crack growth prior to peak load when compared to the unreinforced systems (Fig. 8). Thus LEFM concepts are no longer applicable, and for these materials methods that account for plasticity and/or stable crack growth should be used. Resistance curves (R curve, ASTM E 561-81) are used to characterize the resistance to fracture during slow stable crack extension in such materials. Unlike brittle fracture that is characterized by a single value of fracture toughness (K_{IC}), the resistance curve provides a toughness record as a crack is driven stably by an increasing applied load. These resistance curves (K_R − Δa curve) should be unique for the material and the thickness of interest; and should be independent of crack length, specimen width and specimen type for through-the-thickness cracks. The resistance

Table II **Critical Stress Intensity Factor (MN m$^{-3/2}$) for 14% Polyester Mortar**

	Initial Notch Depth		CMOD Method	
System	Mean	Std Dev	Mean	Std Dev
Polyester mortar	1.00	0.25	1.20	0.15
(nos. of specimens)	(20)		(10)	

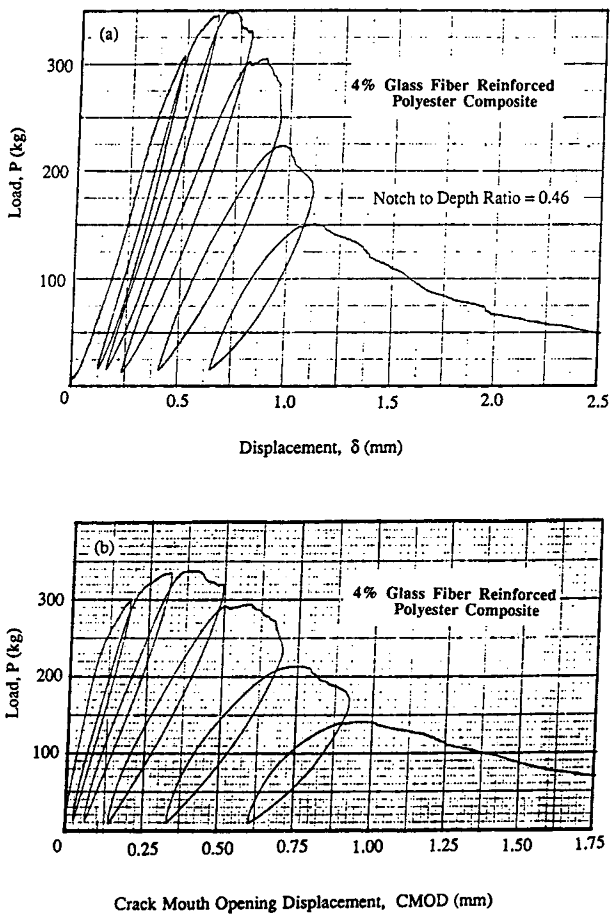

Figure 8 Typical response of 4% glass fiber reinforced 18% polyester composite: (a) load–displacement; (b) load–CMOD.

curve can then be used to predict the stable crack growth and instability (or maximum) load for the cracked structural component.

To construct the K_R curve, effective crack length based on the CMOD method was used. Once the load (P) and the corresponding effective crack length is known from the test records the value of K_I can be obtained from eq. (2a). The variation of K_R with crack extension (Δa) is shown in Figure 8(a) and it is observed that the K_R variation with Δa seem to lie in a narrow band. The K_R–Δa relationship is expressed in the form

$$K_R = K_0 + \rho(\Delta a)^n \tag{6}$$

where K_0, ρ, and n are parameters obtained from least square fit of the data as suggested by Newman.[19] It is evident that eq. (6) cannot be linearized. The constant n was varied from zero to two in steps of 0.01 and the corresponding value of K_0 and r for every iteration was obtained by least square analysis. The value of n which furnished the minimum error [eq. (7), Fig. 9(b)] was selected with the corresponding K_0 and ρ. In this least square procedure an arbitrary weighting factor of $\Delta a^{1/2}$ was used. This weighting factor was used because of difficulties in determining the initiation of crack extension. Using this weight factor, the stress intensity factors at small values of Δa will have less weight than displacements at large Δa. With the value of n assumed, the sum of squares of the error is given by

$$\sum_{p=1}^{P} e_m^2 = \sum_{p=1}^{P} (K_p - K_0 - \rho(\Delta a)p^n)^2 \Delta a_p \tag{7}$$

where p is the number of data points (Δa_p, K_p). Figure 9(b) shows the variation of square of the error with n and the error is minimum when $n = 1.0$. The values of K_0, ρ, and n are 1.87 MN $m^{-3/2}$, 0.33, and 1.0 respectively. Hence the curve is linear and in Figure 9(a) the predicted K_R–Δa curve [eq. (6)] is compared to the experimental data. The data points (K_I, Δa) obtained beyond the peak load show larger scatter and greater deviation from the K_R–Δa relationship.

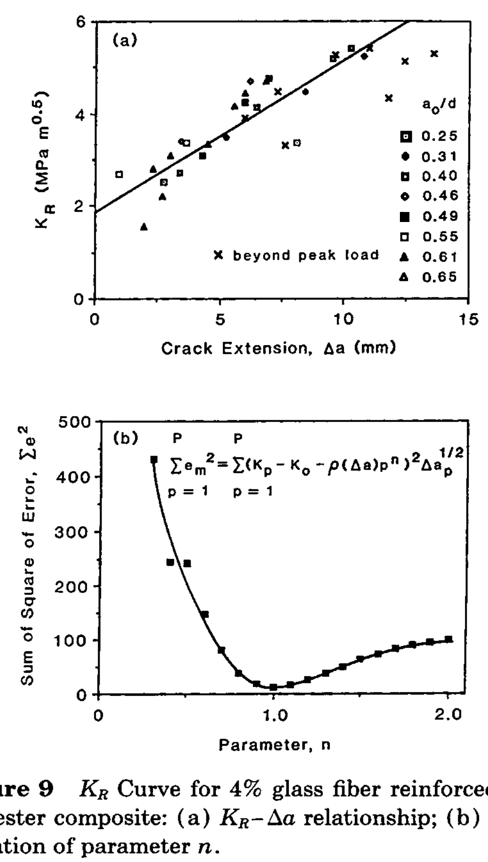

Figure 9 K_R Curve for 4% glass fiber reinforced 18% polyester composite: (a) K_R–Δa relationship; (b) determination of parameter n.

CONCLUSIONS

Based on the complementary experimental and analytical study on polyester composites the following conclusion can be advanced.

1. The flexural strength and critical stress intensity factor of polyester mortar increases with increasing polymer content. The flexural modulus goes through a maximum with increase in polymer content. At room temperature the critical stress intensity factor is linearly related to the flexural strength of the polyester mortar.
2. The critical stress intensity factor of polyester mortar is independent of crack length for the range of crack lengths investigated in this study and satisfy the ASTM E 399 requirements for plain-strain conditions.
3. The critical stress intensity factor of the 14% polyester mortar determined from effective crack length concepts do show an increase in value when compared to the initial notch depth calculations. There is a 5–15% crack extension during pre-peak loading of the mortar specimens. The critical stress intensity factor for the polyester mortar is independent of the initial notch depth.
4. The fracture resistance curves (K_R curves) based on stress intensity factor has been developed for the 4% glass fiber reinforced polyester composite.

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