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TECHNICAL PAPER

Effect of Notch Root Radius on the Fracture Toughness of Epoxy and 0.5 wt% Amino MWCNTs-Reinforced Nanocomposite

K. Chandra Shekar[1] · B. Anjaneya Prasad[2] · N. Eswara Prasad[3]

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Abstract Significantly improved fracture toughness can be imparted by introducing amino multi-walled carbon nanotubes to neat epoxy resin. The present work reports on the fracture toughness evaluation of neat epoxy resin and its nanocomposite. Both the materials with varied finite notch root radii ρ, (in the range of 110–750 μm) are subjected to mode-I (tensile) fracture. The study reveals that the critical notch root radius (below which the apparent fracture toughness K_{IQ} does not vary with ρ) of neat epoxy is 340 μm and the same for nanocomposite is 300 μm.

Keywords Nanocomposite · MWCNTs · Mode-I fracture toughness · Critical notch root radius

1 Introduction

Unique atomic structure, enormous aspect ratio and extraordinary mechanical, electrical, thermal and magnetic properties make carbon nanotubes (CNTs) as ideal reinforcement candidates in nanocomposites [1, 2]. A pre-existing varied finite notch root radii specimens is subjected to mode-I loading. Mode-I deformation often takes place when a symmetric cracked specimen is subjected to pure bending or pure tensile loading conditions.

The specific aim of the present study is to evaluate the effect of notch root radius on the fracture toughness of epoxy/nanocomposite under mode-I loading conditions. Previous studies on monolithic materials, polymer matrix composites and metal matrix composites have shown that the initiation fracture toughness is independent of the notch root radius, ρ, below a critical value designated as ρ_o, because of the blunting that occurs before the crack extends. Beyond this critical value, the fracture toughness increases linearly with ρ [3–8]. Thus the critical radius, ρ_o, provides insight into both the intrinsic fracture and triaxially constrained deformation characteristics of the materials as well as help in selecting notch root radius for subsequent evaluation of fracture resistance where fatigue pre cracking is not feasible [5]. The effect of finite notch root radius is evaluated and reported in the present study, in case of neat epoxy and nanocomposite. The scanning electron microscopy (SEM) was also used to examine the fracture surfaces to correlate the fracture mechanisms to the fracture toughness values evaluated participating in the fracture of the epoxy/nanocomposites.

2 Experimental

2.1 Processing of Material

Diglycidyl ether of bisphenol-A (DGEBA) epoxy resin and diethyl toluene diamene (DETDA) hardener were used as matrix system. Both epoxy and hardener were purchased from M/s Fine finish, Mumbai. Amino-functionalized MWCNTs used in this study were purchased from M/s Chemapal industries, Mumbai. The MWCNTs synthesized by the chemical vapor deposition (CVD) process had a

✉ K. Chandra Shekar
kcs_chandra2003@yahoo.co.in

1 Department of Mechanical Engineering, Vignan Institute of Technology and Science, Deshmukhi, Hyderabad 508 284, India

2 Department of Mechanical Engineering, Jawaharlal Nehru Technological University College of Engineering, Hyderabad 500 085, India

3 Regional Centre for Military Airworthiness (Materials), CEMILAC, DRDO, PO Kanchanbagh, Hyderabad 500 058, India

diameter of 20–30 nm and lengths of 2–4 μm. Nanocomposites consisting of an epoxy matrix loaded with 0.5 wt% amino MWCNTs were prepared. Initially, amino-functionalized MWCNTs resin mixture was made by mixing a 0.5 wt% of amino-functionalized MWCNTs to epoxy resin. Dispersion of the amino-functionalized MWCNTs was ensured by sonication with a probe type sonicator (Mesonix-3000, USA) for 45 min followed by ball milling (Insmart Systems, India) for 180 min at 250 rpm. Hardener in the ratio of 1:4 of the resin weight was added to the resin mixture followed by ball milling at same rpm. To realize nanocomposites, resin mixture was poured into a die and cured for 2 h at 120 °C followed by 3 h curing at 180 °C, which is the peak cure temperature of the composition under study. Neat epoxy specimens (without nanotube loading) were fabricated simply mixing the curing agent with the resin under stirring and casting the specimens in an oven following an identical cycle as for the nanocomposites.

2.2 Specimen Preparation

Mode-I fracture toughness specimens of both the materials pertain to the geometry of single edge notch bend specimen (SENB) as described in ASTM standard D5045-99 with nominal dimensions of L = 60 mm (with an included span length equivalent to 4 times the width), W = 8 mm and B = 4 mm maintaining W/B of 2. The notches with different notch root radii were introduced by means of Isomet low speed cutting saw using 150 μm and 300 μm thick diamond wafer blades by progressive cutting. Notch root radius measurements were made with the help of a profile projector.

2.3 Fracture Toughness Testing

The mode-I fracture toughness i.e., plane strain fracture toughness was determined using the three point bend specimens according to ASTM standard D5045-99. The tests were conducted at ambient temperature and in the laboratory air atmosphere using SENB specimen. The fracture tests were performed at 0.5 mm/min loading rate using an universal testing machine (United, Model STM 50 KN, USA). For each type of material, minimum five specimens were tested. The load versus crack opening displacement data were recorded as per the ASTM standard D5045-99. The crack extension was monitored using unloading compliance technique/method. The conditional or apparent plane strain fracture toughness (K_{IQ}) was calculated using the following equations:

$$K_{IQ} = \left(\frac{P_Q}{BW^{1/2}}\right) f(x) \quad (1)$$

where $0 < x < 1$ and x = a/W, where "a" is crack length and W is specimen width and,

$$f(x) = \frac{6x^{1/2}[1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]}{(1+2x)(1-x)^{3/2}} \quad (2)$$

$$B, a, W - a \geq 2.5\left(\frac{K_{Ic}}{\sigma_y}\right)^2 \quad (3)$$

where K_{IQ} is conditional fracture toughness, f the shape factor, P_Q the peak load, B the specimen thickness, and σ_y the yield strength.

3 Results and Discussion

3.1 Notch Profiles

Notches of fixed length maintaining a crack length to width ratio of 0.45–0.55 and varying notch root radius (ρ) were introduced. Notches of higher notch root radius were also introduced by the same Isomet by progressive cutting. The notches thus introduced were found to have finite root radius (ρ) typically of the orders varying from 110 to 750 μm. The ρ values were determined experimentally using Delta TM 35 x–y profile projector. The variation of ρ for a particular isomet blade cutting was found to be less than 10 μm. In homogenous materials the ideal cutting behavior is expected to result in ρ = 0.5–0.55T, where T is the thickness of isomet blade. The crack lengths were maintained within the range of 0.45–0.55 times the specimen width in both the materials, as per the ASTM standard D5045-99 for the determination of K_{Ic} (when $\rho < \rho_o$) or K_{IQ} (when $\rho > \rho_o$) values.

3.2 Load–Displacement Data

The load–displacement data for both neat epoxy and nanocomposite specimens are shown in the Figs. 1 and 2, respectively. These load–displacement data for the two materials are obtained for the specimens with different notch root radii (ρ). The load–displacement diagrams for all the specimens are linear up to the fracture load, which shows the brittle and linear elastic behaviour of the tested materials. It may be noted that the peak load in each experiment was considered as the fracture load. The data regarding the notch root radius and specimen dimensions along with the obtained fracture toughness are given in Tables 1 and 2. Absolute values of crack length (a), specimen width (W) are given along with normalized values of a/W.

3.3 Effect of Notch Root Radius

The fracture toughness of the materials are evaluated and analyzed by investigating the influence of varying notch

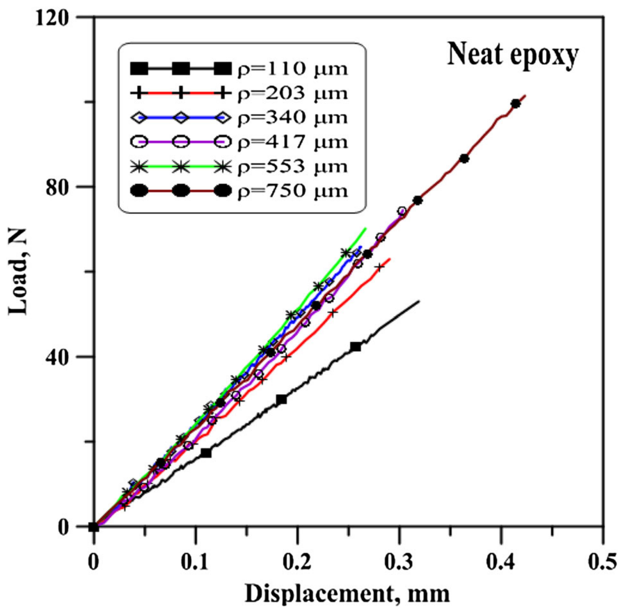

Fig. 1 Load–displacement data obtained for neat epoxy with varying finite notch root radius (110–750 μm)

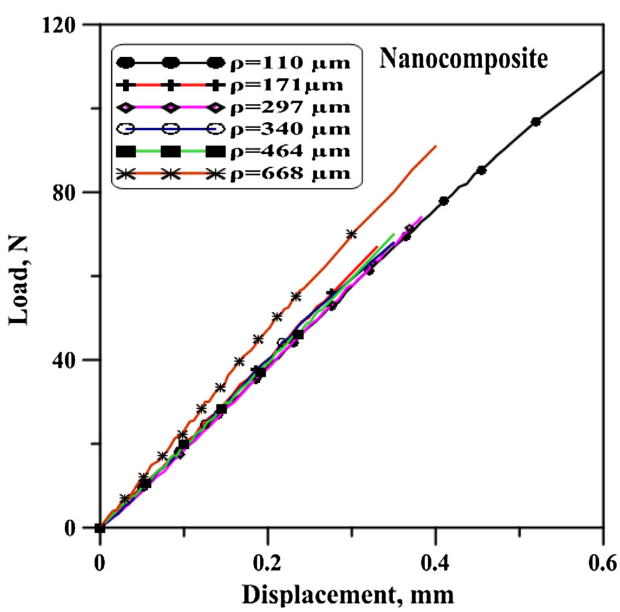

Fig. 2 Load–displacement data obtained for nanocomposite with varying finite notch root radius (110–668 μm)

root radii in both the materials. The actual fracture toughness of the material leads to overestimate, if the notch root radius is too large. It is therefore necessary to control the notch root radius and to machine it carefully in order to have a notch root radius below the sensitivity threshold of the material. The apparent fracture toughness as a function of the notch root radius for both neat epoxy and nanocomposite is summarized and shown in Table 1. It can be clearly seen that specimens with lower notch root radius

Table 1 Effect of notch root radius on apparent fracture toughness of neat epoxy and nanocomposite specimens

Specimen no.	Thickness of isomet blade used (μm)	Notch root radius (ρ) (μm)	$\sqrt{\rho}$ ($\sqrt{\mu m}$)	Fracture toughness, K_{IQ} (MPa$\sqrt{m}$)
Neat epoxy				
1	150	110	10.49	1.673
2	150	203	14.26	1.703
3	300	340	18.43	1.698
4	300	417	20.42	2.185
5	300	553	23.52	2.699
6	300	750	27.39	3.304
Nanocomposite				
1	150	110	10.49	1.978
2	150	171	13.08	2.052
3	150	297	17.23	1.959
4	300	340	18.45	2.250
5	300	464	21.53	2.914
6	300	668	25.83	3.530

exhibits lower fracture toughness (ρ = 110 μm) and the value increases with increase in the notch root radius. The presence of sharper notch facilitates the easy propagation of the crack which results in lesser amount of energy absorption. The results of both neat epoxy and nanocomposite show similar trend. The higher energy absorbed in ρ > 110 μm is due to crack extension from a relatively blunt initial crack. The notch sensitivity was extensively studied and reported [9–13]. But similar studies in case of neat epoxy and nanocomposite are not reported till date. In such cases, crack extension was found to take place under critical strain controlled behavior, the variation of K_{IQ} with the square root of notch root radius should be linear. This is verified with the data obtained in the present study for both neat epoxy and nanocomposite materials illustrated in Fig. 3a, b, respectively. The plane strain fracture toughness in nanocomposite is higher than that in neat epoxy. This is due to the presence of amino MWCNTs which is evident from the SEM micrographs shown in the Fig. 4a, b. The increased plane strain fracture toughness can be attributed to the improved dispersion of amino MWCNTs in the epoxy matrix and better interfacial reactions between the functional groups on the carbon nanotube surfaces and the polymer matrix (Fig. 5). The fracture surfaces of both the neat epoxy and nanocomposite were investigated by field emission scanning electron microscope in order to study the fracture mechanisms. As shown in Fig. 4a the fracture surface of neat epoxy is relatively smoother, which shows a low energy fracture as compared to nanocomposite. Furthermore, the surface roughness increases with amino MWCNTs addition suggesting that the crack propagation

Table 2 Apparent fracture toughness data (K_Q, MPa$\sqrt{}$m) of the neat epoxy and nanocomposite specimens

Specimen no.	W (mm)	B (mm)	a (mm)	a/W	f(a/W)	P_Q (N)	P_{max} (N)	K_{IQ} (MPa$\sqrt{}$m)	K_{max} (MPa$\sqrt{}$m)
Neat epoxy (σ_f = 100 MPa)									
1	8.14	4.04	4.23	0.52	11.35	53	53	1.673	1.673
2	8.25	4.17	4.12	0.50	10.65	60	60	1.703	1.703
3	8.24	4.02	3.87	0.47	9.70	64	64	1.698	1.698
4	8.20	4.18	3.73	0.51	10.99	74	74	2.164	2.185
5	8.24	4.02	4.12	0.50	10.65	92	92	2.685	2.699
6	8.16	4.14	4.41	0.54	12.14	101	101	3.301	3.304
Nanocomposite (σ_f = 125 MPa)									
1	8.45	3.60	4.04	0.48	10.01	68	68	1.860	2.052
2	8.40	3.50	4.03	0.48	9.99	73	73	2.250	2.250
3	8.45	3.40	3.80	0.45	9.14	67	67	1.959	1.959
4	8.50	3.55	3.91	0.46	9.41	70	70	1.976	1.978
5	8.30	3.42	3.98	0.48	9.98	91	91	2.914	2.914
6	8.27	3.12	3.63	0.45	9.14	109	109	3.570	3.530

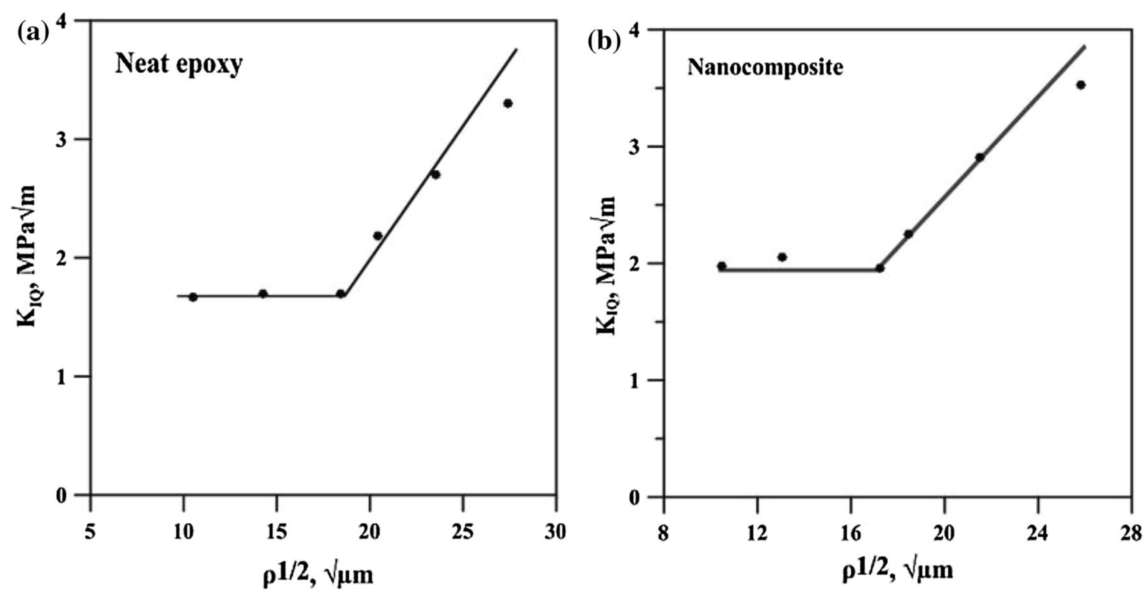

Fig. 3 Variation of apparent fracture toughness with square root of notch root radius for **a** neat epoxy and **b** nanocomposite

in the nanocomposite is opposed by rigid and stiff amino MWCNTs. It can also be seen that a linear relationship between K_{IQ} and ρ is obtained for both the materials reaching a critical value of notch root radius (ρ_o) below which the value of apparent fracture toughness is invariant (Fig. 3a, b). This invariant K_{IQ} is the material fracture toughness (provided the other condition of validity B ≥ 2.5 $(K_{IQ}/\sigma_f)^2$, where K_{IQ} is the apparent fracture toughness and σ_f is flexural strength, with an assumption that fracture stress scales with flexural strength). This critical value of notch root radius (ρ_o) thus determined is approximately equal to 340 μm in case of neat epoxy and 300 μm in case of nanocomposite. This implies that in both the materials, if the notch root radius is below the critical value then the fracture toughness is solely a material property conditional to the fact that the criteria for linear elastic fracture mechanics (LEFM) and plane strain are satisfied.

3.4 LEFM Controlled Fracture

Among various fracture criteria, a material fails under linear elastic conditions and the controlling fracture criteria is under linear elastic fracture mechanics (LEFM) [14], if the following conditions are fulfilled.

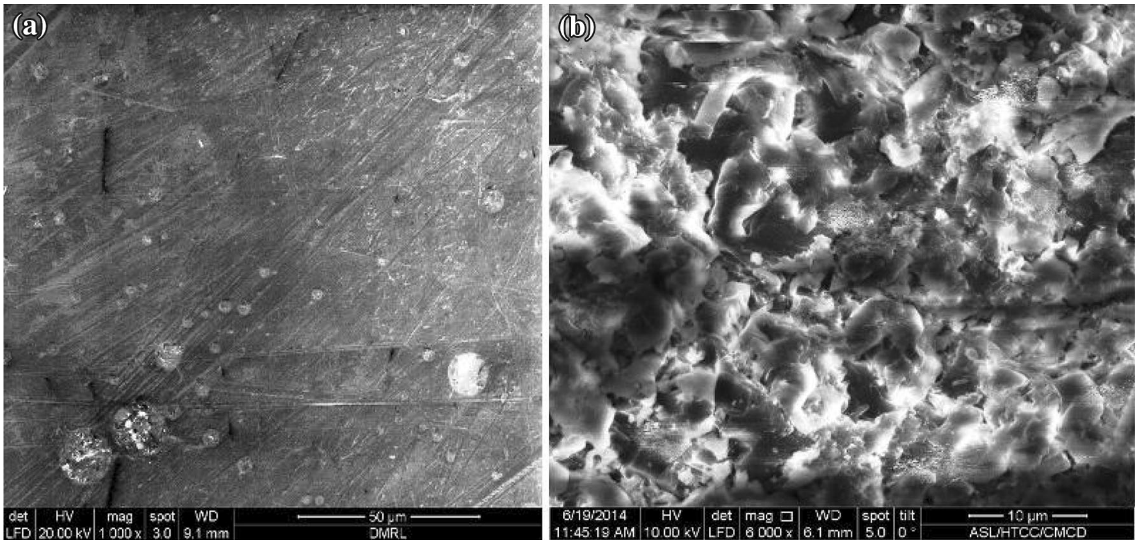

Fig. 4 FESEM micrographs of mode- I fracture surfaces for: **a** neat epoxy, **b** nanocomposite, both the materials show quasi-cleavage fracture, with higher energy fracture in case of nanocomposite

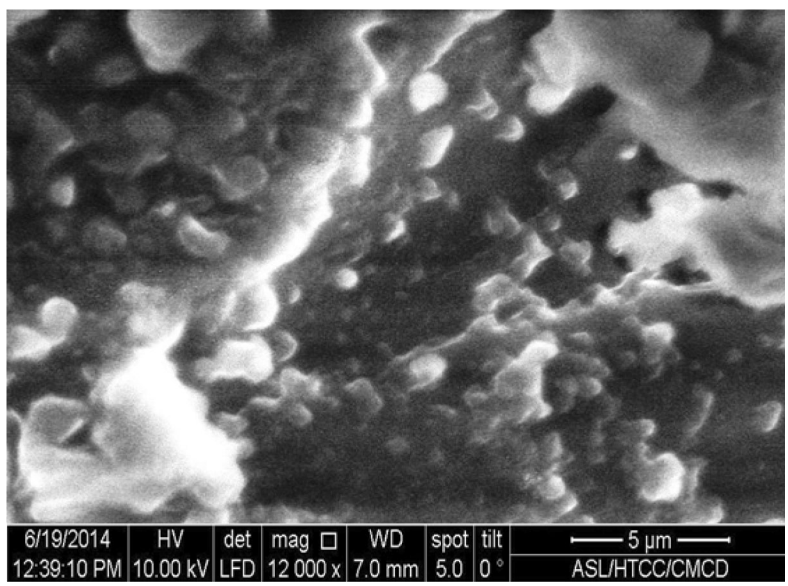

Fig. 5 Distribution of MWCNTs in epoxy matrix

(i)

$$\text{Thickness criterion}: \quad B \geq 2.5\left(\frac{K_{Ic}}{\sigma_{ys}}\right)^2 \tag{4}$$

(ii)

Uncracked ligament length criterion :

$$b, W - a \geq 2.5\left(\frac{K_{Ic}}{\sigma_{ys}}\right)^2 \tag{5}$$

And most importantly

(iii)

$$\text{If } \left(\frac{K_{Ic}}{\sigma_{ys}}\right) = constant, \quad \text{in all cases of fracture} \tag{6a}$$

$$\text{or } K_{Ic} \propto \sigma_{ys} \tag{6b}$$

The valid K_{Ic} values of neat epoxy and nanocomposite are examined and the above criteria are fulfilled. It is found that both the materials satisfy all the three above conditions (see the data in Table 3).

Table 3 LEFM fracture properties in neat epoxy and nanocomposite

S. no.	Thickness (B), (mm)		Un cracked ligament length (b) (mm)		$\frac{(K_{Ic})_{Avg}}{(\sigma_y)_{Avg}}$ ($\sqrt{m}$)
	B (Actual)	Minimum 'B' for LEFM $\left[B = 2.5\left(\frac{K_{Ic}}{\sigma_{ys}}\right)^2\right]$	b (Actual)	Minimum 'b' for LEFM $\left[b = 2.5\left(\frac{K_{Ic}}{\sigma_{ys}}\right)^2\right]$	
Neat epoxy					
1	4.04	0.70	3.91	0.70	0.017
2	4.17	0.73	4.13	0.73	
3	4.02	0.72	4.37	0.72	
4	4.18	1.19	4.47	1.19	
5	4.02	1.82	4.12	1.82	
6	4.14	2.73	3.75	2.73	
Nanocomposite					
1	3.60	0.67	4.41	0.67	0.016
2	3.50	0.81	4.37	0.81	
3	3.40	0.61	4.65	0.61	
4	3.55	0.63	4.59	0.63	
5	3.42	1.36	4.32	1.36	
6	3.12	1.99	4.64	1.99	

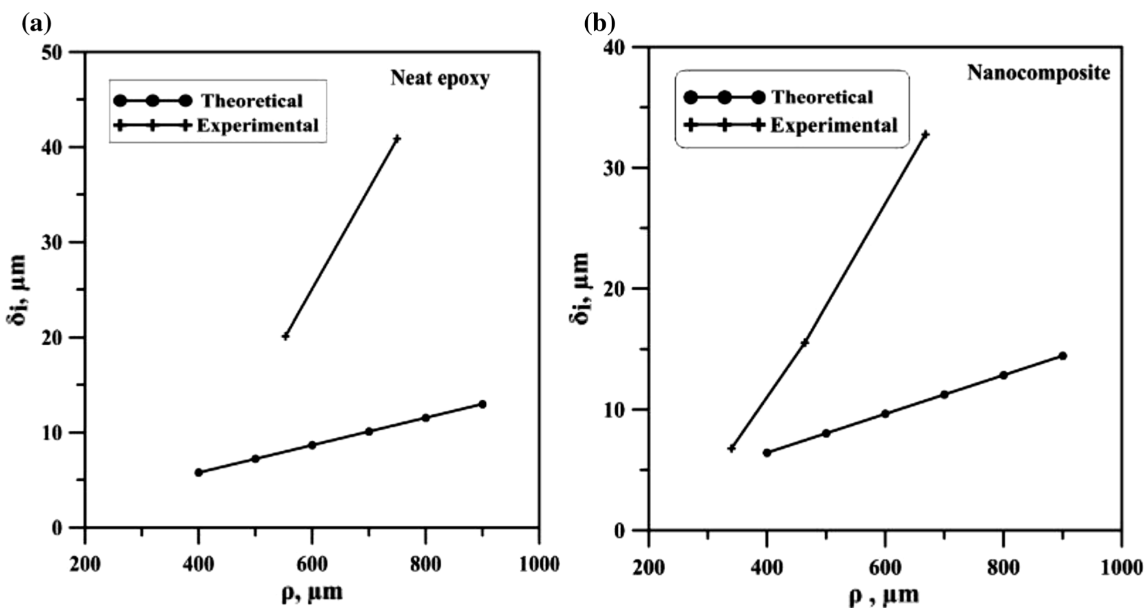

Fig. 6 Variation of crack opening displacement at crack initiation with root radius in **a** neat epoxy and **b** nanocomposite

Now, if K_{Ic} is found to be directly proportional to the σ_f, it can be surmised that the fracture resistance of both the materials scales with increase in strength properties. This is further corroborated from the fact that the ratio of K_{Ic}/σ_f is almost the same for both the materials (0.017 $\sqrt{m}$ for neat epoxy, 0.016 $\sqrt{m}$ for nanocomposite; see the data in Table 3).

3.5 Strain Controlled Fracture

In order to establish if critical strain is the controlling fracture feature/characteristic in these materials, a fracture criterion suggested by Crowe and Gray [8] is used. According to them, if there is a linear relationship between δ_i (crack opening displacement or crack tip opening displacement) and ρ for $\rho \geq \rho_o$, then the fracture is strain controlled. Such δ_i at crack initiation can be calculated using the relation [8]:

$$\delta_i = \frac{0.49K_{Ic}^2}{\rho_o E\sigma}, \text{ for } \rho > \rho_o \quad (7)$$

where K_{Ic} is the fracture toughness, E is the Young's modulus (2886 and 3319 MPa for Neat epoxy and Nanocomposite materials, respectively), σ is the fracture strength (100 and 125 MPa for Neat epoxy and Nanocomposite materials, respectively) and ρ_o is the critical notch root radius (340 µm for neat epoxy is and 300 µm for nanocomposite). δ_i versus ρ plots for neat epoxy and nanocomposite materials are shown in Fig. 6a, b, respectively. It can be seen that in both the cases the behavior is linear. In neat epoxy the experimentally measured slope is 0.1054 while the theoretical slope is 0.0144. However, there exists a linear relationship between displacement at the crack initiation point and the notch root radius. There is nearly an order of disparity between the experimentally measured and theoretically estimated slopes of the linear fit. Similarly in nanocomposite material, the experimentally measured slope is 0.07977 which does not agree with the theoretical slope obtained using the same Eq. 7 which is 0.0160. The disagreement between the theoretical and experimentally measured slopes in both the neat epoxy and nanocomposite may be attributed to small scale yielding or nonlinear deformation. Insignificant extent of non linear deformation or small scale yielding for both materials were attributed to failure by LEFM. Such large difference in the theoretical and experimentally found CTOD and ρ_o values can be attributed to a grossly varied local elastic modulus values which can be substantially different at local regions as compared to the assumed Young's modulus values due to inhomogeneous dispersion/bonding of MWCNTs and matrix epoxy.

The above observations imply that the fracture in both the materials is strain controlled. The plots represent the linearity between the displacement at the crack initiation and the notch root radius (greater than the critical notch root radius) and thus provide the evidence that the fracture occurred in the materials is strain controlled. In nanocomposite the higher fracture toughness value is due to the presence of amino MWCNTs.

4 Conclusions

The present experimental investigation has led to the following specific conclusions.

1. The fracture toughness is independent of the notch root radius below a critical notch radius (ρ_o) of 300 µm for neat epoxy and increases linearly with the square root of notch root radius for ρ greater than 300 µm. Similarly, the ρ_o for nanocomposite is 340 µm.
2. The fracture toughness of the neat epoxy is nearly 1.7 MPa$\sqrt{m}$, whereas the same is nearly 2.02 MPa$\sqrt{m}$ for nanocomposite. The increase ($\approx$20 %) in fracture resistance is due to the presence of amino MWCNTs in neat epoxy.
3. Using the fracture mechanics approach suggested by Crowe and Gray, it is established that the fracture process in both the materials is strain controlled.

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