Scaling of Fatigue Crack Growth in Pristine Epoxy

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Paper Number: 10

July 2, 2018

ABSTRACT

This work proposes an investigation on the scaling of fatigue crack growth in pristine epoxy. Towards this end, fatigue fracture tests on geometrically scaled Single Edge Notch Bending (SENB) specimens were conducted.

It is shown that Paris-Erdogan law exhibits strong size effect on both slope and threshold for pure epoxy. This indicates that the fatigue Fracture Process Zone (FPZ) size is significantly larger compared to the quasi-static one. In fact, fracture tests conducted by Mefford *et al.* [1] and a comprehensive literature study [2] showed that the FPZ has a negligible effect on the scaling of the fracturing behavior of pure epoxy under quasi-static loading condition.

By introducing a fatigue size effect model based on the energetic-equivalence framework, the thresholds in the Paris-Erdogan curves representing geomatically-scaled specimens can be successfully adjusted whereas the slopes still exhibit size effect. This latter aspect needs to be studied further to enable the application of the Paris-Erdogan law to quasi-brittle structures of different sizes and geometries.

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INTRODUCTION

The fatigue fracturing behavior of polymer nanocomposites has been studied in the literature by means of a well-known Paris-Erdogan law. However, an aspect often overlooked is the fatigue behavior on the scaling of nanocomposites. This is an important aspect since, for Paris-Erdogan law [3], the fatigue crack rate as a function of stress intensity factor (SIF) amplitude is based on Linear Elastic Fracture Mechanics (LEFM) which neglects the effects of Practure Process Zone (FPZ) and treats it to be a mathematical point. According to the comprehensive studies on the fracturing behavior of nanocomposites reported by Mefford *et al.* [1] and Qiao *et al.* [2] as well as several other theoretical studies [4–10], the effects of FPZ on nanocomposites are generally not negligible. Thus, accounting for the effects of FPZ in Paris-Erdogan law is an aspect of utmost importance.

This work proposes an investigation on the scaling of fatigue crack growth in pristine epoxy, which paves the path for investigating graphene nanocomposite in the future work. It is shown that, even for pristine epoxy, there is a noticeable size effect on the coefficients of Paris-Erdogan law. This is not the case for pristine epoxy under quasi-static loading conditions. This indicates that the cyclic FPZ size is presumably larger than the monotonic FPZ size thus leading to a size dependent Paris-Erdogan law.

MATERIALS AND METHODOLOGY

Materials

The thermoset polymer used for all of the tested specimens was composed by an EPIKOTETM Resin MGSTM and an EPIKOTETM Curing Agent MGSTM RIMH 134-RIMH 137 (Hexion [11]) combined in a 100:32 ratio (by weight).

Specimen Preparation

In order to study the scaling of fatigue fracturing behavior, as illustrated in Figure 1, the geometically scaled Single Edge Notch Bending (SENB) specimens were designed based on ASTM D5045-99 [12]. The epoxy and hardener were manually mixed for 10 minutes, degassed for 20 minutes by using a Vacmobile vacuum system [13] and poured into silicone molds made of RTV silicone from TAP Plastics [14] to create geometrically scaled specimens with consistent sizes.

After curing, the specimens were pre-cracked through a two stage process. The first step consisted in creating a notch about one-fifth of the specimen width by means of a 0.2 mm wide diamond coated saw. Then, tapping was preferred to sawing to create the last portion of the crack in order to provide a very sharp crack (about 4 μm). The initial crack length was about 0.25-0.4D where D is the specimen width.

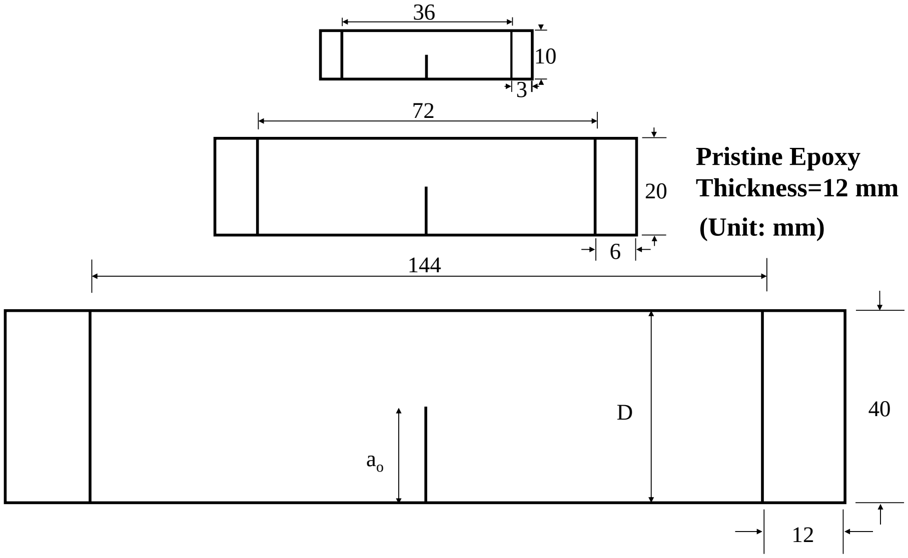

Figure 1. Geometry of Single Edge Notch Bending (SENB) Specimens. Units: mm.

Testing

Fatigue fracture tests on Single Edge Notch Bending (SENB) specimens were performed on a Servo-hydraulic 8511 Instron machine with the capacity of 20 KN. The loading conditions are the followings: (1) a sinusoidal ramp load; (2) load ratio $R = 0.1$; (3) load frequencies were 5 Hz, 7.4 Hz and 11 Hz for $D = 10$ mm, 20 mm and 40 mm respectively in order to have the roughly same strain rate for specimens with different sizes.

EXPERIMENTAL RESULTS

Fatigue Crack Growth

Thanks to the transparence of pure epoxy, the evolution of crack growth of specimens during fatigue tests can be monitored by a digital microscope. Based on this direct measurement of crack length, the growth of normalized crack length with respect to number of cycles can be plotted in a semi-logarithmic coordinate as shown in Figure 2. The normalized crack length was defined as $\alpha = a/D$ where a is the crack length.

It is worth mentioning here that the crack propagation of pure epoxy under fatigue loading has the following three stages: (1) uneven crack front due to the tapping propagates into an even crack front; (2) even crack front propagates into a parabolic shape of crack front; (3) stable propagation of the crack with almost the same parabolic shape. The chord length of the parabolic shape crack is about 0.8 mm, 0.9 mm and 1.1 mm for $D = 10$ mm, 20 mm and 40 mm respectively.

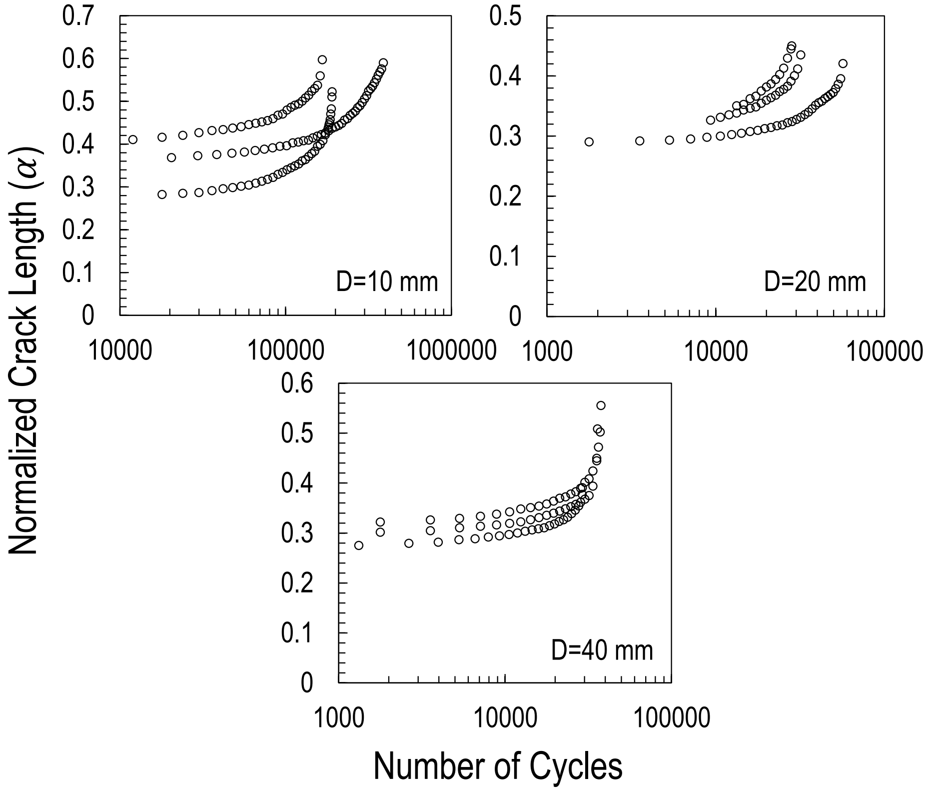

Figure 2. Normalized crack length vs. cycles for pure epoxy.

Paris-Erdogan Curves

Following Linear Elastic Fracture Mechanics (LEFM), the Mode I Stress Intensity Factor K has the following form:

$$K = \sqrt{E^{*}G(\alpha)} \tag{1}$$

where E^{*} is Youngs Modulus for plane strain condition, υ is Poisson's ratio and $G(\alpha)$ is the energy release rate which equals to $\sigma_N^2 D g(\alpha_0)/E^{*}$. In this expression, $\sigma_N = 3PL/2tD^2$ and $g(\alpha_0)$ is the dimensionless energy release rate which can be found from Mefford *et al.* [1]. The relationship between crack growth rate (da/dN) and mode I Stress Intensity Factor (SIF) amplitude $(\Delta K = (1 - R)K)$ for all the investigated specimen sizes can be plotted in double logarithmic coordinates as illustrated in Figure 3. The experimental data in Figure 3 are usually referred to the following well-known equation (Paris-Erdogan law):

$$da/dN = C(\Delta K)^n \tag{2}$$

As can be noted from Figure 3, three aspects of Paris-Erdogan curves are affected by the non-negligible Fracture Process Zone (FPZ): (1) initial crack growth rate; (2) the slope n of curve; (3) fatigue threshold (K_{th}) and critical stress intensity factor amplitude ΔK_c.

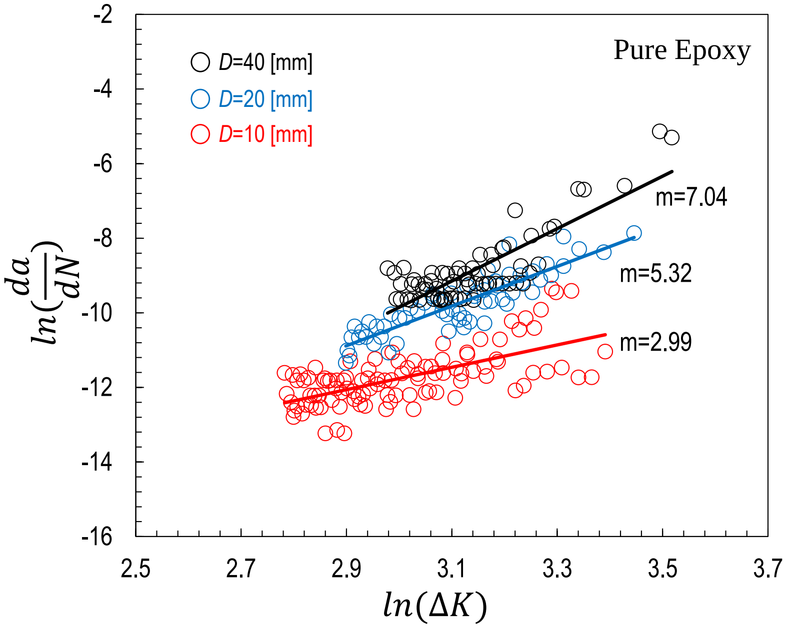

Figure 3. Relationship between crack growth rate and SIF amplitude for pure epoxy.

SEM Analysis

In order to investigate the main mechanisms of fatigue crack growth in the scaling of pristine epoxy, the SENB specimens were cut and the fracture surfaces were gold-coated in order to be used for Scanning Electron Microscopy (SEM) by a JSM-6010PLUS/LA Electron Microscope [15]. The SEM images of some samples representing different sizes are showed in Figure 4.

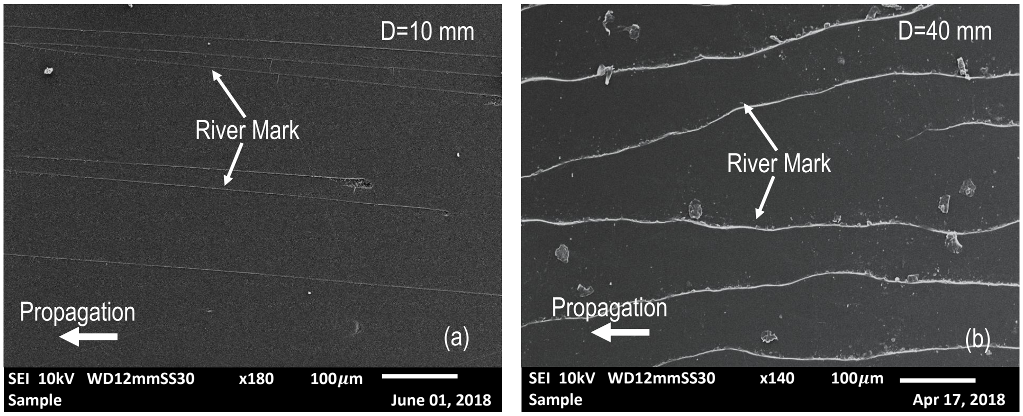

Figure 4. Fracture surfaces of specimens: (a) $D = 10$ mm; (b) $D = 40$ mm.

As can be noted from Figure 4, river marks parallel to the crack propagation direction were observed for all the investigated specimen sizes. This can be explained by the fact that crack front propagates on the different planes. Contrary to the fact that beach marks perpendicular to the crack propagation direction features the fatigue fracture morphology in metals. In addition to this, river marks are more pronounced for large specimen while small specimen has less pronounced river marks.

Analysis and Discussion

According to the previous studies on the scaling of fatigue crack growth in sandstone with a theoretical model describing the experimentally observed size effect in Paris-Erdogan curves [16], it is considered that during each load cycle the energy dissipation associated with the growth of the macrocrack is equal to the sum of the energy dissipations associated with the propagation of all the active nanoscale cracks inside the cyclic FPZ. This can be written in the following:

$$U_{c,\infty} da/dN = \sum_{i=1}^{n_a} U_a da_i/dN \tag{3}$$

where $U_{c,\infty}$ =critical energy dissipation per unit growth of the macrocrack in an infinitely large specimen, U_a =critical energy dissipation associated with the breakage of one atomic bond. a_i =length of ith nanoscale crack and n_a =number of active nanoscale cracks in the cyclic FPZ.

By considering a well-estiblished transition state thoery from Kramer [18], the fatigue growth rate of a crack at the nanoscale can be obtained in the following:

$$da_i/dN = \upsilon_i e^{-Q_0/kT} \Delta K_{ai}^2 \tag{4}$$

where υ_i =constant determined by the geometry of the nanoscale element, Q_0 =dominant free activation energy barrier, k =Boltzmann constant, T =absolute temperature and ΔK_{ai} =SIF amplitude of the nanoscale element.

With the assumption on the average energy dissipations associated with the nanocrack growth and dimensional analysis on the number of active nanoscale cracks in the cyclic FPZ, the fatigue growth rate of a macro crack in a finite specimen size can be written in the following form:

$$da/dN = \frac{A\Delta K^2}{U_c} \Phi(\Delta K^2/EU_c) \tag{5}$$

where $A = \omega^2 U_a \upsilon_a e^{-Q_0/kT}$, U_c =critical energy dissipation per unit growth of the macrocrack in a finite specimen and ΔK =SIF amplitude of a macro crack. By borrowing the ideal on the Size Effect Law (SEL) of quasi-static loading to the cyclic loading scenario: $U_c = U_{c,\infty}[D/(D + D_{0c})]$ where D_{0c} can be considered as transitional size for cyclic loading, the following equation can be obtained:

$$da/dN = \frac{A\Delta K_D^2}{U_{c,\infty}} \Phi(\Delta K_D^2/EU_{c,\infty}) \tag{6}$$

If it is assumed that function Φ is self-similar which means that $\Phi(\Delta K_D^2/EU_{c,\infty}) = \Delta K_D^{2q}/E^q U_{c,\infty}^q$. By leveraging this, eq.(6) can be further written as the following form:

$$da/dN = C\Delta K_D^n \tag{7}$$

where $C = AE^{1-n/2}U_{c,\infty}^{-n/2}$, $n = 2+2q$, $\Delta K_D = (1+D_{0c}/D)^{0.5}\Delta K$ =size adjusted SIF amplitude.

After implementing Eq.(7) into the experimental data obtained from fatigue fracture tests, as illustrated in Figure 5, the size effect on the fatigue threshold (K_{th}) and critical stress intensity factor amplitude ΔK_c are removed. To achieve this, cyclic FPZ size is roughly about 1 mm, 3 times larger than the monotonic FPZ found by Mefford *et al.* [1]. However, size effect on the slope and initial crack growth rate still exist in the adjusted Paris Erdogan law.

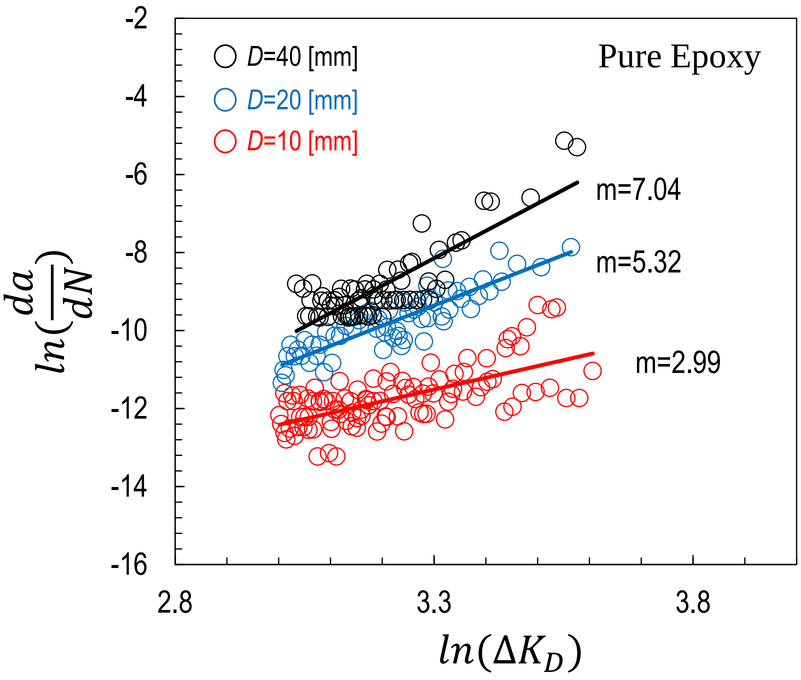

Figure 5. Size-adjusted Paris-Erdogan curves by Eq.7

CONCLUSION

According to the forgoing analysis on the scaling of fatigue crack growth in pristine epoxy, the following conclusions can be elaborated:

1. Fatigue crack growth of pure epoxy has a three-stage process: (1) uneven crack front propagates into even crack crack front; (2) even crack front propagates into a parabolic shape of crack front; (3) stable crack propagation with almost the same parabolic shape;

2. Fatigue tests on the geometrically scaled Single Edge Notch Bending (SENB) pure epoxy specimens indicate that there is an appreciable size effect on the Paris-Erdogan law coefficient C, exponent n and inital crack growth rate.

3. This phenomenon is not the case for pure epoxy under quasi-static loading which exhibits the negligible size effect on the scaling of fracturing behavior due to the fact that Fracture Process Zone (FPZ) size is relatively small compared to the investigated sizes reported by Mefford *et al.* [1]. This pronounced size effect on the scaling of fatigue crack growth in pure epoxy indicates that the cyclic FPZ size is

presumably larger than the monotoic FPZ size;

4. By introducing a fatigue size effect model based on the energetic-equivalence framework [17], the coefficient C can be adjusted to be independent with specimen sizes which turns out that the cyclic FPZ size is roughly about 3 times lareger than the monotonic FPZ size. However, the size effect on the slope in Paris-Erdogan curve of pure epoxy can not be adjusted due to the fact that this model does not take size effect on the exponent n into consideraton.

Future work will focus on investigating the size effect on exponent n in order to have a size independent Paris-Erdogan law. In addition to this, the scaling of fatigue crack growth in graphene nanocomposite will be studied in the future in order to understand the fatigue fracturing behavior on the scaling of nanocmoposites which has potential applications in many fields.

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