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An investigation on Mode II fracture toughness enhancement of epoxy adhesive using graphene nanoplatelets

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Abstract

Epoxy adhesive has a great potential use in different areas which may encounter impact loadings, the fracture behavior of epoxy adhesive needs to be improved to meet the safety requirement. This paper experimentally studies the dynamic mode II fracture toughness of an epoxy adhesive reinforced by different content of graphene nanoplatelets (GNPs) with compliance-based beam method (CBBM). Typical R-curves of the neat epoxy adhesive and nanocomposites with different graphene content under the loading rate of 2m/s are obtained. From the experimental results, the dynamic critical strain energy release rate of nanocomposites increases compared with the value of neat epoxy indicating the effectiveness of graphene on dynamic mode II fracture toughness improvement. The dynamic mode II fracture toughness of nanocomposites reinforced by 0.5wt% GNP exhibit a 41% enhancement compared with neat epoxy adhesive, while no further increase was observed when the nanocomposites loaded the GNP content of 0.75%.

Key Words: Mode II fracture toughness; impact condition; graphene nanoplatelets/epoxy adhesive

1 Introduction

Epoxy adhesive has been widely used to bond metal components, fiber-reinforced composites (FRP), and concrete structures [1–5]. It has shown many advantages in saving structural weight, reducing stress concentration and corrossions

[6–11]. However, the brittle nature of epoxy adhesive significantly reduces its service life and limits the potential use in many applications. In open literatures, a lot of efforts have been made in improving the fracture toughness of epoxy adhesives, but most works still cannot meet the requirements in the industries especially when the structures withstand impact loadings.

Recently, nanofillers have attracted significant interest due to their effectiveness in improving the basic mechanical properties and fracture toughness of epoxy adhesives [12]. Many types of fillers such as metal particles [13, 14] and carbon fillers including carbon nanofibers (CNFs) [15,16], carbon nanotubes (CNTs) [17,18], and graphene [19,20,21] have been studied. Multi-walled carbon nanotubes (MWCNTs) were added to an epoxy to improve the fracture toughness. Both mode I and mode II fracture toughness of epoxy increased with the addition of MWCNT into epoxy [22].

Compared with other types of fillers, graphene exhibits much better performances, due to its exceptional mechanical behavior and large aspect ratio. Hitherto, graphene reinforced nanocomposites have been extensively studied to for improving mechanical and other functional properties [23–25].GNPs were used to enhance the fracture toughness of E-glass/epoxy composites, including modes I, II and III interlaminar fracture toughness. The experimental results showed that the interlaminar fracture toughness was significantly improved under mode I fracture, but not as much for mode II and mode III [26]. Compared with mode I, the mode II fracture toughness of GNPs/epoxy composites are rarely studied, especially when the nanocomposites subjected to impact loadings.

In this paper, GNPs have been used to reinforce epoxy adhesive at different content of 0.5 wt% and 0.75 wt%. The End-Notched Flexure (ENF) specimens with different content of nanocomposites under the loading rate of 2m/s were used to test dynamic mode II critical strain energy release rate of nanocomposites. The variation trend of dynamic mode II fracture toughness with the GNP content were obtained.

2 Experiment

2.1 Materials

The epoxy adhesive used in this paper was manufactured by Kangda Company in Shanghai, a two-component adhesive which contains component A for epoxy and component B for curing agent. This adhesive is commonly used in the construction area for repairing the existing structures and the cure scheme of this adhesive is at room temperature (RT) for 72 h according to the manufacturer of adhesive. The GNPs were fabricated by thermally expanding the graphite intercalated compound (GIC), and the detailed manufacture process can be found in our previous works [19].

2.2 Preparation of GNP/epoxy composites

The dispersion of GNPs in acetone was obtained by sonication for 6 h in a bath sonicator at a graphene concentration of 2mg/ml. Then the dispersions were mixed with a certain amount of component A of the epoxy adhesive, according to the graphene content of the final composites. GNPs and component A of the adhesive were pre-mixed at 2000 rpm for 3h with a magnetic stirrer to evaporate acetone at RT. The temperature of the mixture was then increased to 100 °C for full evaporation of acetone. After cooling down to RT, curing agent (component B of the adhesive) at a stoichiometric ratio was added into the mixture and a planetary mixer (ZYMC-180V,

ZYE Technology Co., Ltd) was used to mix the composites at 2000 rpm for 3 min to obtain the final GNP/epoxy composites. Nanocomposites containing two different graphene contents, including 0.5 wt% and 0.75 wt% were prepared.

2.3 Sample fabrication

The mode II fracture toughness of the adhesive with different content of GNPs was tested using the ENF specimens and the dimensions were shown in Fig.1. Stainless steel with the width of 12mm was used for the adherends. The ENF surfaces were first degreased with acetone to scrub the metal oxide and oil stain, followed by blasting with #60 sandpaper to increase the roughness of the bonding surface. The bonded surfaces were then degreased with acetone and then soaked in a sodium hydroxide solution with a concentration of 20% for 30 minutes. After soaking, it was washed with acetone and distilled water. To control the thickness of GNP/epoxy adhesive as 0.2 mm, two spacers, each with the thickness of 0.1mm, were inserted between the adherends before the application of adhesive. The spacers were removed after the ENF joints being cured. A sharp pre-crack with the length of 70mm was fabricated by a 40 μm thick polytetrafluoroethylene (PTFE) film. The pre-crack was placed in the middle of the spaces to ensure that the pre-crack positions in the mid-plane of adhesive. After curing, scrape the excess adhesive on the side of ENF specimens to complete specimen preparation.

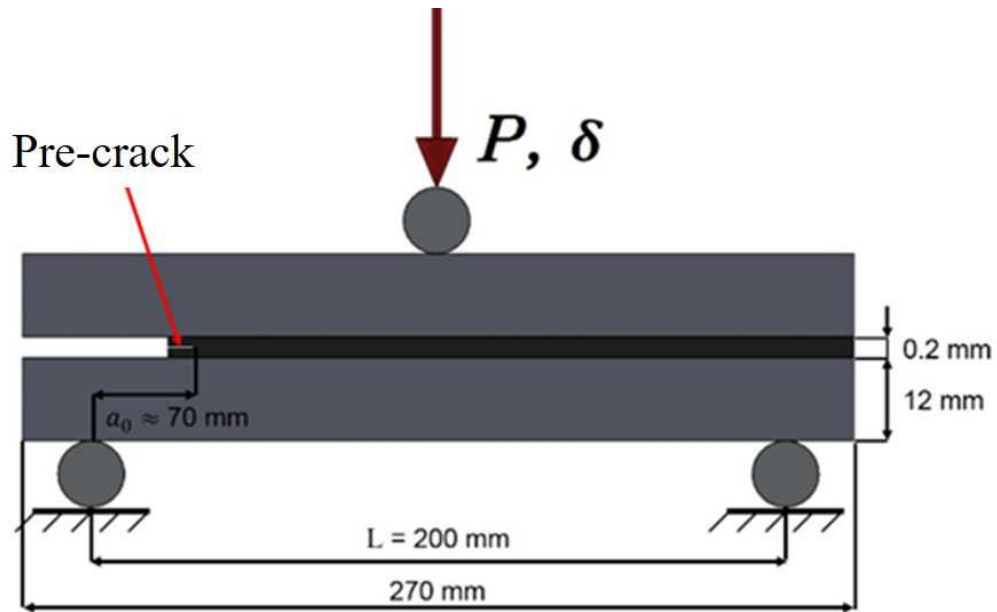


Fig.1. Illustration of ENF specimen dimensions

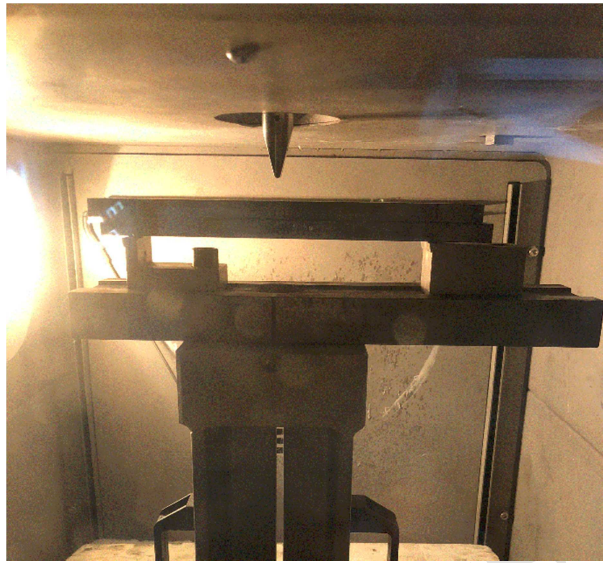
2.4 Test procedure and data analysis method

The ENF specimens under the impact loading of 2 m/s were tested through a drop-weight impact testing machine (INSTRON 9350), as shown in Fig.2.

All the ENF specimens were loaded before the experiment starting until the cracks were extended forward by 2-3 mm on the basis of the pre-cracks to avoid blunted pre-cracks. Force-displacement curves were recorded during the experiment. At least three specimens were experimentally tested for each GNP content.



(a) Impact machine for ENF specimens



(b) ENF specimens placed in the impact machine

Fig.2. Experimental set up for ENF tests under high strain rate at low temperatures

A data analysis method, compliance-based beam method (CBBM), was proposed to obtain the mode II fracture toughness of adhesive [28-30]. It does not need the observation of crack propagation and only the compliance of specimens is used to calculate the mode II fracture toughness of adhesive, which significantly reduces the difficulty of the experiments. This method has been compared with conventional methods which require crack propagation measurement [28,29], and a good agreement is achieved. In this paper, mode II fracture toughness of the neat epoxy adhesive and adhesive with different GNP content under the impact loading speed of 2m/s is obtained through CBBM.

The mode II fracture toughness of adhesive through CBBM is calculated through the Equation (1) to Equation (5) and the implication of the symbol in the Equation (1-5) are listed in Table 1.

The dynamic mode II fracture toughness of the adhesive obtained by CBBM is related to initial compliance, force, the compliance of specimen throughout the entire loading process, especially the compliance after the peak load. The dynamic mode II fracture toughness of epoxy adhesive containing different GNP content is related to all the above parameters.

$$G_{II} = \frac{9P^2 a_{eq}^2}{16B^2 E_f h^3} \quad (1)$$

$$a_{eq} = \left[\frac{C_c}{C_{0c}} a_0^3 + \left(\frac{C_c}{C_{0c}} - 1 \right) \frac{2L^3}{3} \right]^{1/3} \quad (2)$$

$$E_f = \frac{3a_0^3 + 2L^3}{8Bh^3 C_{0c}} \quad (3)$$

$$C_c = C - \frac{3L}{10BhG} \quad (4)$$

$$C_{0c} = C_0 - \frac{3L}{10BhG} \quad (5)$$

Table 1 Implication of the symbol in Equation (1-5)

Symbol	Implication
G_{II}	Mode II strain energy release rate
P	Load
a_{eq}	Equivalent crack length
B	Specimen Width
E_f	Equivalent flexural modulus
h	Specimen Height
C_c	Corrected compliance of specimen
C_{0c}	Corrected initial compliance of specimen
L	half length of the specimen between supports
G	shear modulus of adherend
a_0	initial crack length

3 Results

In order to study the effect of GNP content on dynamic mode II fracture toughness of adhesive, experimental tests on ENF specimens with neat epoxy and different content of GNP/epoxy adhesive under the impact loadings were carried out. Typical force-displacement curves of ENF specimens with neat adhesive and GNP/epoxy nanocomposites with two different graphene contents were shown from Fig.3 to Fig.5.

It can be observed that under the impact loadings, ENF specimens with nanocomposites showed a little bit higher peak load when the GNP content was 0.5 wt% while the peak loading decreased compared with the value for neat epoxy when GNP content continued to increase to 0.75 wt%.

The typical R-curves for neat epoxy and nanocomposites with different GNP content were shown in Fig. 6 and the variation trend of dynamic mode II critical strain energy release rate of nanocomposites with the graphene content was shown in Fig.7.

It can be seen from Fig. 6 and Fig.7 that the enhancement of dynamic mode II fracture toughness was obvious when the GNP added into epoxy adhesive. The nanocomposite reinforced at a graphene content of 0.5 wt% increased by 41% in dynamic mode II fracture toughness compared with the neat epoxy. However, no further increase was observed when the graphene content continued to increase. The dynamic mode II fracture toughness of nanocomposites with GNP content of 0.75 wt% decreased compared with nanocomposites with graphene content of 0.5 wt%, but still showed an improvement of 26% compared with the data of neat epoxy which proved the effectiveness of GNP in enhancing the dynamic mode II fracture toughness of this

epoxy construction adhesive.

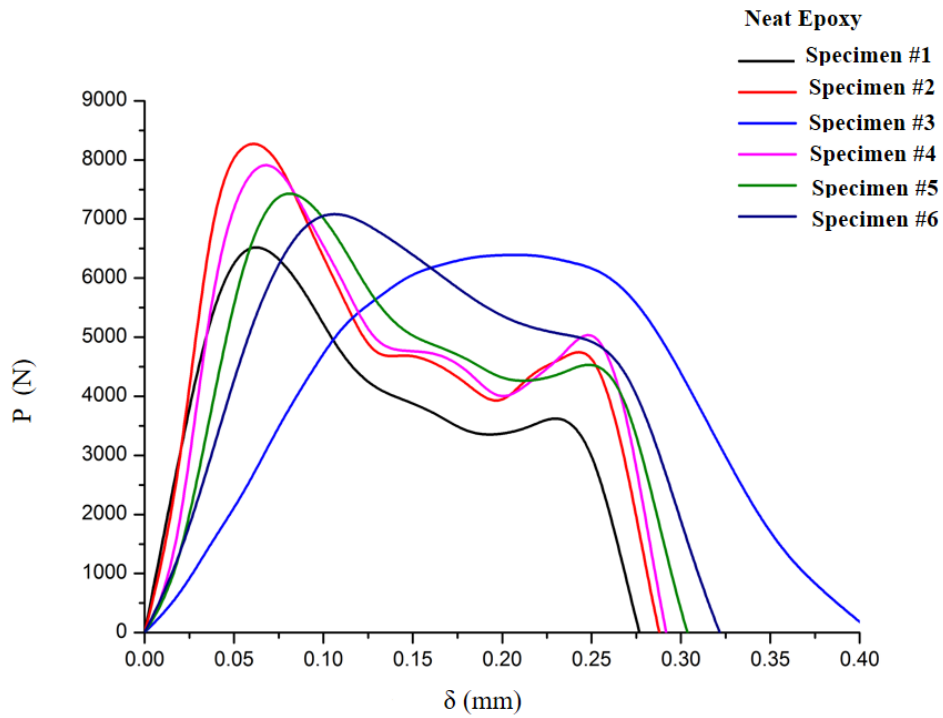


Fig. 3 Force-displacement curves of ENF specimens with neat epoxy

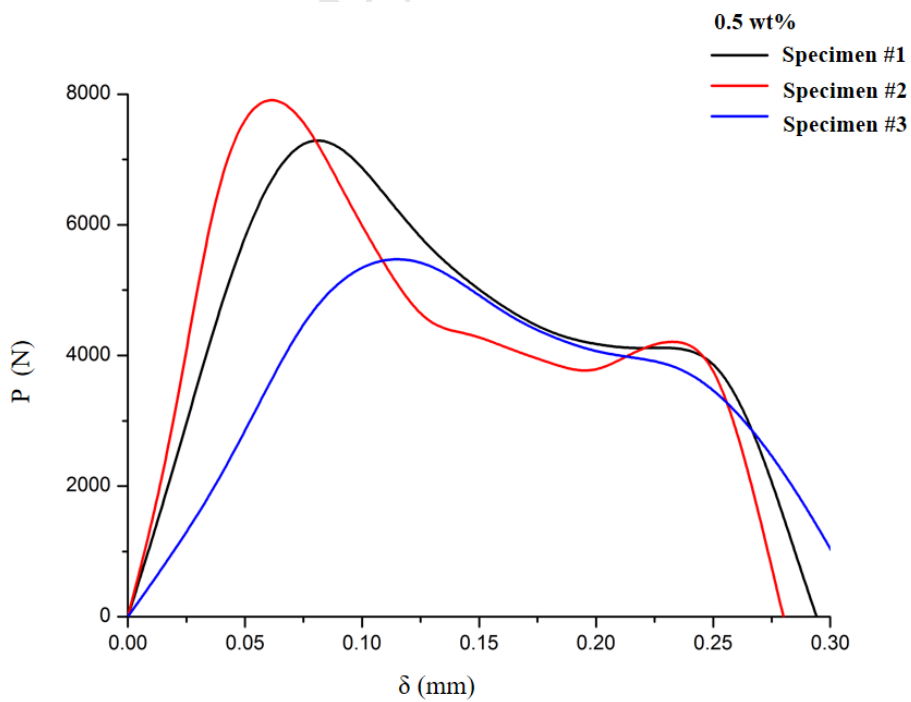


Fig. 4 Force-displacement curves of ENF specimens with epoxy adhesive at a GNP content of 0.5 wt%

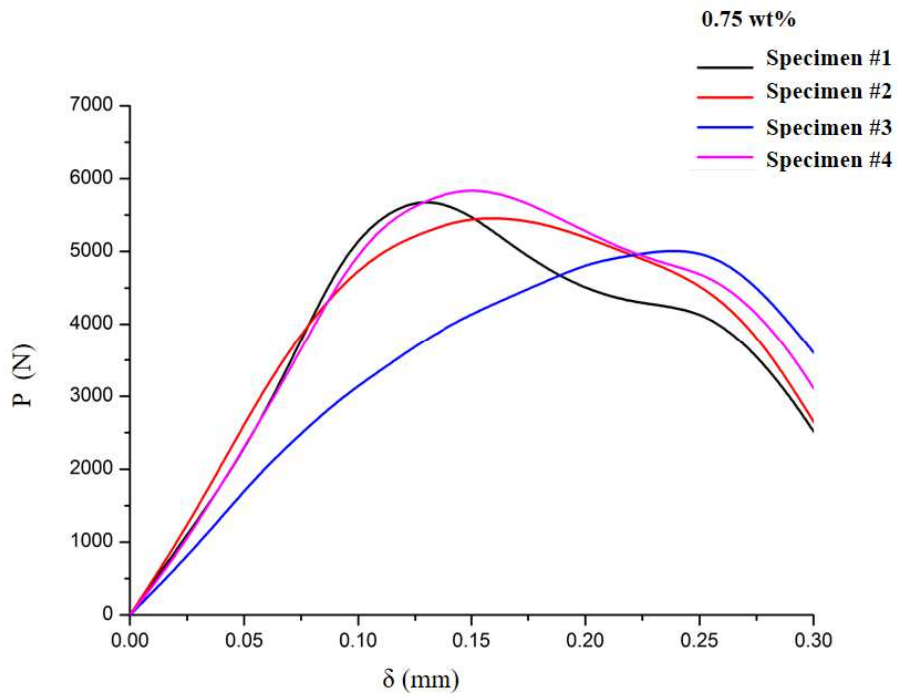
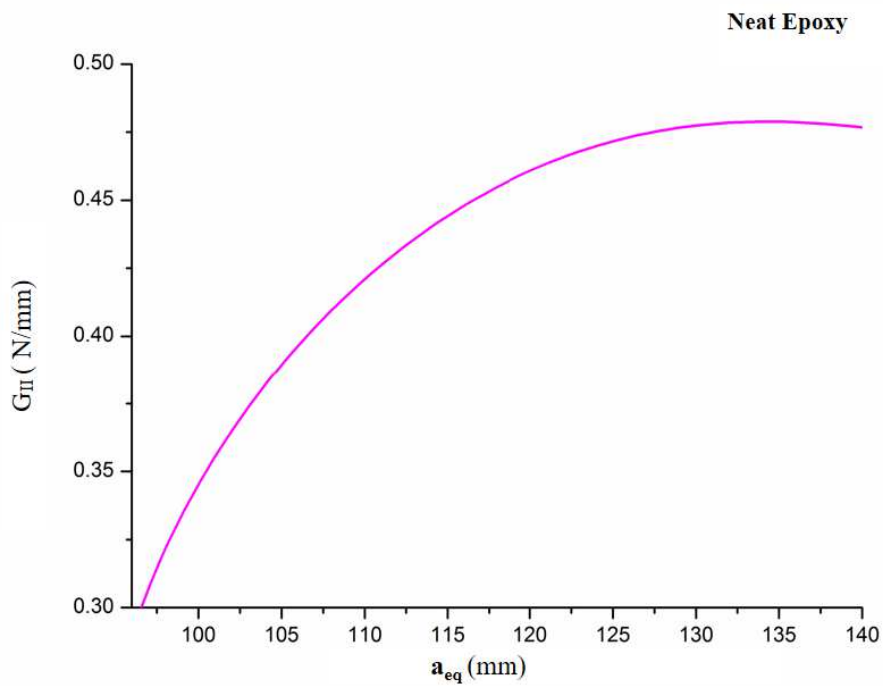
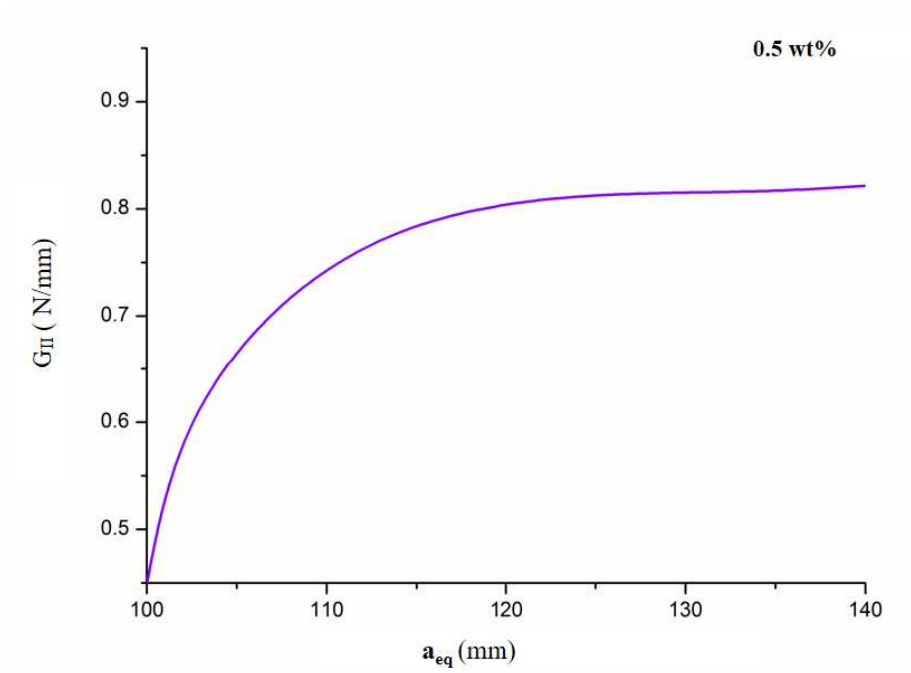


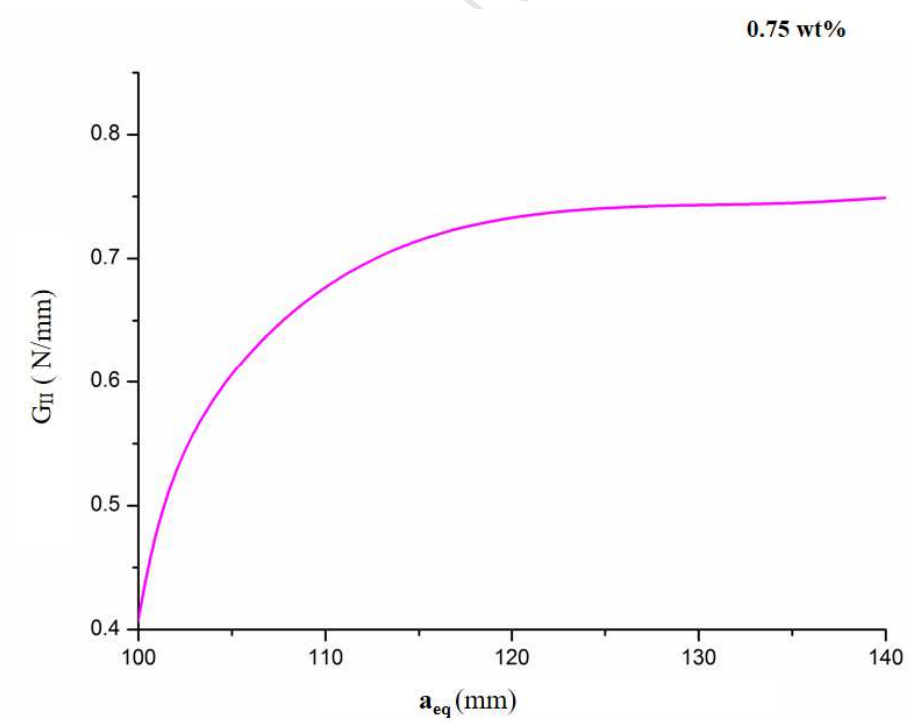
Fig. 5 Force-displacement curves of ENF specimens with epoxy adhesive at a GNP content of 0.75 wt%



(a) Neat Epoxy



(b) 0.5wt%



(c) 0.75wt%

Fig.6 Typical R-curves of DCB specimens containing neat epoxy and different

content of GNP/epoxy nanocomposites.

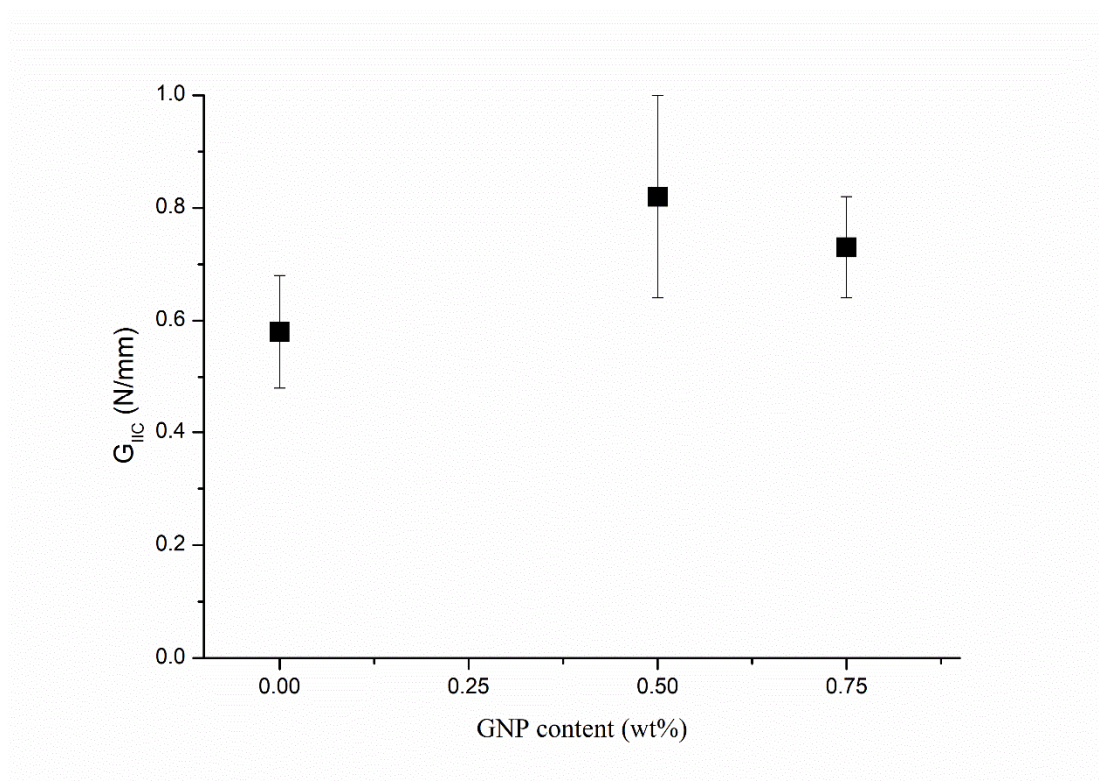


Fig. 7 Dynamic Mode II fracture toughness of nanocomposites with a function of graphene content

4 Conclusion

The dynamic mode II fracture toughness of epoxy adhesive with different graphene content under the loading rate of 2 m/s was experimentally studied. Typical R-curves of the nanocomposites were obtained. From the experimental results, the dynamic critical strain energy release rate of nanocomposites increased compared with the value of neat epoxy which demonstrated the effectiveness of graphene in improving the dynamic mode II fracture toughness for this epoxy adhesive. The dynamic mode II fracture toughness of nanocomposites reinforced by 0.5wt% GNP showed an increase of 41% compared with neat epoxy adhesive, while no further increase was observed when the nanocomposites loaded the GNP content of 0.75%.

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