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Fracture Development and Deformation Behavior of Zeolite-Filled High Density Polyethylene Annealed Composites in the Plane Stress Fracture

This study explores the zeolite-high-density polyethylene (HDPE) composites annealed at 90C which was investigated to obtain the crack growth and deformation behavior under tensile loading until the specimen was completely broken. Crack growth and deformation were obtained from the double-edge-notched tensile (DENT) test. At each stage of fracture development, the value of fracture energy consumed was determined based on the energy partitioning concept. The deformation is assessed at each stage of the fracture process both at the crack initiation and crack propagation. The results describe that the crack grows in a similar way, and the deformation behavior involved varies with the essential work of fracture (EWF) values for each stage of crack growth.

Keywords: *crack development, deformation behavior, high density polyethylene, zeolite, crack behavior.*

1. INTRODUCTION

The resistance of the material fracture is characterized by appropriate fracture mechanics, which is a principal appliance in enhancing the mechanical achievement [1,2]. In this case, fractography becomes very prominent to be used to understand deeper the causes of fracture. It can even be used to tell in advance the hypothetical failures due to its capability to predict the crack growth. In terms of materials fracture analysis, their behavior is often arduous to ascertain due to their high adherence and nonlinearity. In addition, their deformation phenomena do not match the fracture mechanics as required in linear elastic fracture mechanics.

The material toughness value according to EWF concept is regarded as the energy consumed in the fracture process zone (FPZ) where a new surface is created. In the case of crack growth due to tensile load, the EWF value can be obtained using a double-edge-notched tensile (DENT) test [3,4], for which the related specimen is shown in Fig. 1. In the plane-stress fracture condition, total energy consumed in a DENT test is composed of two distinct parts: (i) the essential work for the creation of new fracture surface (W_e), and (ii) the work for plastic deformation throughout the ligament part (W_p). The total fracture work (W_f) is the sum of two parts of energy consumption. The W_f can be expressed:

$$W_\mathrm{f} = W_\mathrm{e} + W_\mathrm{p} = w_\mathrm{e}.t.\ \ell + \beta.w_{\mathrm{p}.}.t.\ell^2 \tag{1}$$

where t is specimen thickness, and ℓ is initial ligament length. The specific work of fracture, $w_f \left(= W_f / t.\ell\right)$ can be expressed as:

$$w_f = w_\mathrm{e} + \beta.w_\mathrm{p}.\ell \tag{2}$$

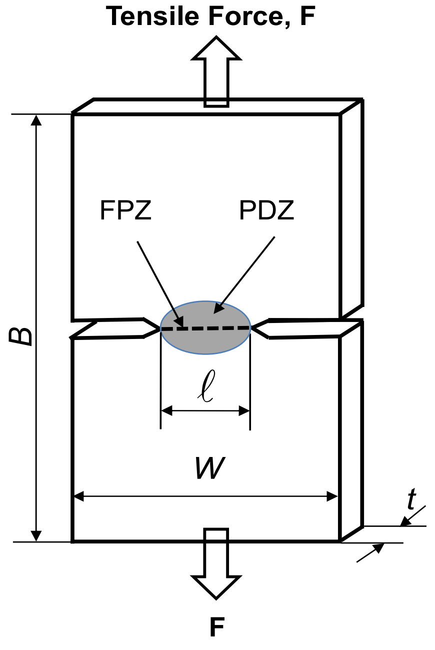

Figure. 1. Schematic diagram of DENT specimen showing of FPZ and plastic deformation zone (PDZ) as well as their typical dimension, B = total length.

To ensure that the fracture occurred in plane stress, the ligament length satisfies the following conditions [3,5].

$$\left(3-5\right) t \le \ell \le \min\left(\frac{W}{3}\ or\ 2r_p\right) \tag{3}$$

where w_e, βw_p, β, and W are the specific EWF, specific non-EWF, a shape factor of the plastic region, and

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specimen width, respectively. The parameter r_p is the plastic zone size which can be estimated as:

$$2r_p = \frac{1\,E\,w_e}{\pi\,\sigma_y^2} \tag{4}$$

where E is Young's modulus and σ_y is the yield stress of materials.

In fracture process, many previous works [6-8] have expressed w_e value in terms of work before and after the deformation of neck formation in FPZ. In many ductile materials, total fracture occurs immediately after the neck is initiated by the instability of neck development during tensile deformation [9]. The increase of composite strength by the strain-hardening is caused by the formation of the neck which cannot compete with the rise in stress due to the decrease in the specimen cross-section. Nevertheless, in the case of ductile materials such as zeolite-reinforced HDPE, the strain-hardening rate in the neck is quick sufficient to offset the reduction in cross-sectional area, so that the process of neck development is stable, and its size is growing at a fixed rate [10]. Previous studies evaluating the EWF of polyethylene such as HDPE [6, 11,12] have neglected the process of neck developing in analyzing the energy needed during the fracture. In contrast to those studies, Jar and co-workers [13,14] have observed neck formation during fracture process and reported that in plane stress fracture of DENT specimen, the neck was initiated along the ligament part, it spread to adjacent areas which had been deformed at an early step of the test.

This work aims to investigate the fractured development and the deformation behavior of DENT specimen of zeolite-filled HDPE annealed composites. An investigation conducted in plane stress condition, associated with the energy consumption during fracture process based on EWF test. The crack development and deformation process during crack growth until total fracture occurred were examined and recorded, the associated energy dissipation, and their specific fracture work values were determined. The criteria for the crack development based on the crack-tip opening angle were discussed.

2. EXPERIMENT METHOD

2.1 Materials and samples preparation

The materials used consist of natural zeolite powder obtained from Malang-Indonesia and HDPE from a commercial-grade provided by PT. Lotte Chemical Titan Nusantara Indonesia. They are the same materials used in previous studies [15-19], as well as the treatment is given to them. The injection-moulded zeolite-HDPE composites with zeolite concentration of 0 wt.%, 2.5 wt.%, 5 wt.%, and 7.5 wt.% were annealed at 90°C for 12 hrs at a heating rate of 1°C/min and cooling rate of 0.5 °C/min. All composite specimens that have been formed are kept out directly sunlight.

2.2 Fracture Test

Fracture testing is performed by referring to the provisions as in the EWF test. The DENT specimens used for EWF tests were subjected to a tensile load until the specimen is totally broken. The test was conducted at the universal testing machine at a constant crosshead speed of 4 mm/min, at ambient temperature. To ensure that the plane-stress fracture occurred, the DENT specimens used as shown in Fig. 1 were prepared to have a width (W) of 50 mm and the initial ligament length (ℓ) ranging from 9 to 15 mm (corresponding to 3-5 times to t), according to suggested value by many researchers [6,11,12].

Crack growth on DENT was observed on both sides and front views of ligament section. Deformation process involved during crack growth until total fracture occurred, such as yielding, necking, and tearing was determined using energy partitioning method proposed by Karger-Kocsis and coworkers [20-24].

As an assumption, the strain of DENT tested in the direction of ligament length was ignored [14, 25]. During the test, the crack growth and deformation process were recorded using SONY HDR-CX405 Handycam.

3. RESULTS AND DISCUSSION

All the DENT specimens tested were totally fractured throughout the tensile test. Type of their load (F)-displacement (x) curve for 2.5 wt.% zeolite-HDPE annealed composite (ℓ = 15 mm) was shown in Fig. 2. Their deformation involved based on the energy partitioning work of fracture [20,21,26] consists of deformation for yielding (δ_y) and necking subsequent tearing (δ_n), as shown in Fig. 2. The elongation of the surface layer (δL) formed after crack coalescence is excluded [27].

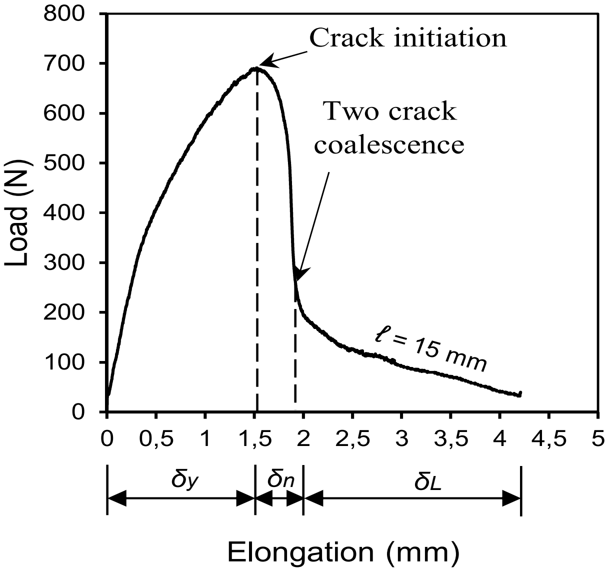

Figure. 2. The F-x curve showing the deformation and crack development in DENT test of 2.5 wt.% zeolite concentration with ℓ = 13 mm. The δy, δn, and δL is the deformation for yielding, necking subsequent tearing, and surface layer, respectively.

The F-x curves of all specimen tested are similar, but different in longitudinal deformation in each stage of fracture process. The self-similarity indicates that the fracture development and deformation behavior phenomenon are appropriately analyzed using the EWF concept. Table 1 shows the deformation of DENT tested showing the δy, δn, and δL for all composites. From

Table 1, it can be seen that the elongation during yielding process tends to increase with increasing the zeolite content, however, the elongation of the composite was more difficult to occur at necking stage as well as a surface layer formed by increasing zeolite concentration in a host matrix. It is due to the particles blocking the host matrix to deform during the composite subjected to the tensile load. As a result, the crack tip undergoes plasticity which causes the composite to fracture in a brittle manner.

Table 1. Elongation of DENT specimens (ℓ = 15 mm)

Zeolite/HDPE	Elongation (mm)		
	Δy	Δn	δL
0/100	14.5	5.1	40.1
2.5/97.5	13.2	4.0	30.4
5/95	16.02	3.4	20.57
7.5/92.5	16.7	3.3	10.0

Plotting *we* and βw_p vs. the zeolite concentration for all specimens tested were shown in Fig. 3. The partitioning fracture work scheme consists of two distinct parts: (i) EWF for yielding ($w_{e,y}$) and necking subsequent tearing ($w_{e,n}$), and (ii) non-EWF for yielding ($\beta w_{p,y}$) and necking subsequent tearing ($\beta w_{p,n}$). The partitioning work of fracture excludes the fracture work of surface layers formed after the crack coalescence was occurred in the mid-thickness region [27,28]. The phenomena shown in Fig. 3 express that fracture energy is not constant during the development of the crack, but is, in fact, different from the deformation mechanism involved in the crack growth process. In the binding and binding process, the EWF decreases by the incorporation of 2.5 wt.% zeolite. However, that value increased in the annealed composite with zeolite concentration of 5 wt.%. The further increase of zeolite content led to a reduction of EWF. However, the non-EWF (βw_p) that is associated with plastic work is carried out on the outside plastic deformation zone decreases by increasing the zeolite concentration in the host matrix.

The earlier process in data deduction has been considerably used for DENT test, which is based on some of the following assumptions in the determination of essential fracture work parameter: (i) plastic zones evolved fully around the ligament region prior to crack growth, (ii) the necking take place within FPZ, and (iii) fracture strain rate in the FPZ is constant during crack growth [11,28-30]. The strain hardening may not be enough to produce stable neck correspond to the later two assumptions.

During the test for DENT of zeolite-HDPE composites, the deformation and fracture process were presented in Fig. 4 (front view of 5 wt.% zeolite-HDPE specimen with ℓ = 15 mm) and Fig. 5 (side view of 5 wt.% zeolite-HDPE specimen with ℓ = 13.5 mm. White arrows indicate the stage of the process. From Fig. 4b, it can be explained that crack was initiated from both notch tips when the maximum load was reached, with the load dropping at an almost constant rate, also shown in Fig. 2.

Nevertheless, the load drops sharply when the two crack tips met each other in the middle section of ligament across the thickness. Moreover, the two cracks fully coalesced shortly after, leaving only the surface layer which still connected the two parts, as described in Fig. 4c and 5d. Furthermore, the increase in displacement lead to elongation, and gradual fracture of the surface layers, as shown in Fig. 4d, 4e, 5e, and 5f.

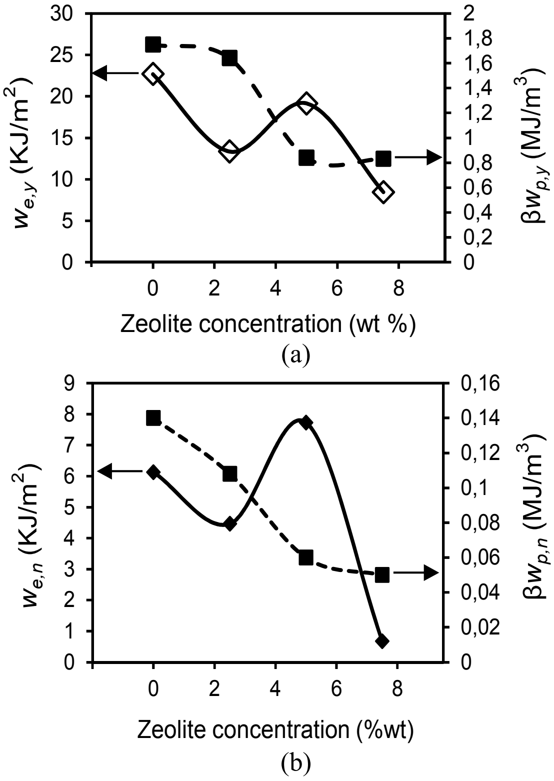

Figure 3. Essential and non-essential energy for yielding (a), and necking subsequent tearing (b).

Figure 4 and 5 also depicted that zeolite-HDPE composites in limited ductility condition, the plastic deformation did not create sufficient strain hardening to stabilize the process of neck development. Figure 4b shows that the crack development is accompanied by the enlargement of the FPZ size, with the size enlarge in the direction of specimen length to be rather slower than that in the ligament length direction.

Since the FPZ length increased as well as the crack growth, the two FPZs met each other at the middle section of the specimen, as depicted in Fig. 4b. The DENT specimens underwent fracture soon after the neck inception stage in which the neck grew from the two notch tips. This cracking mechanism is different from what occurred in high resilient materials that are based on different stability in the process of necking. Plastic zone shape is an attribute to neck propagation in DENT test. It can be elucidated as follows. Strain increment ($d\varepsilon$) in the plastic zone can be obtained from the relationship as follows [14,28,29]:

$$d\varepsilon = \frac{dz}{\gamma L} \tag{3}$$

where dz, γ, and L are displacement increment, the shape factor representing the active plastic zone shape and remaining ℓ, respectively. Strain rate ($d\varepsilon/dt$) in the fracture process zone depends on only the γ value due to the cross-head speed (dz/dt) was constant during the test.

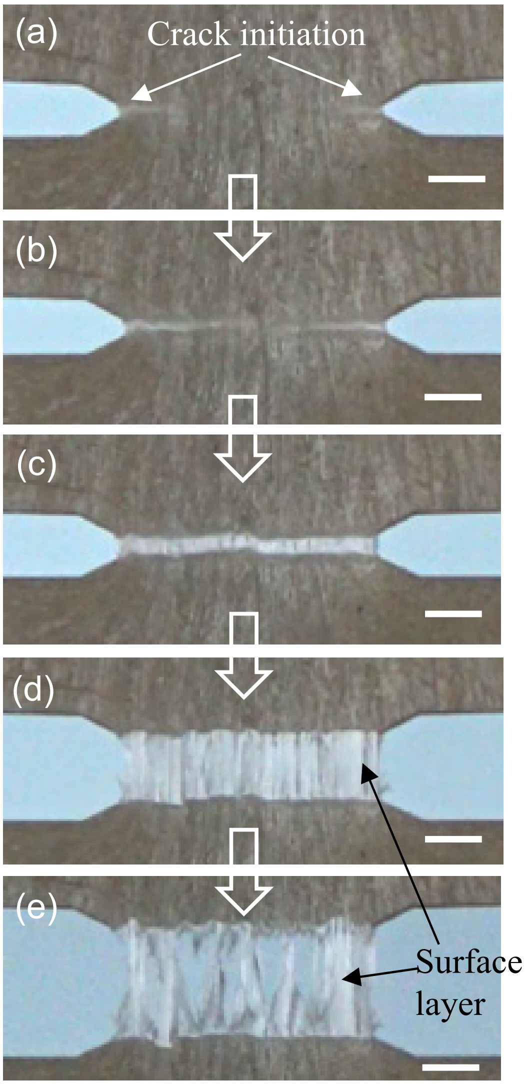

Figure 4. The fracture development and deformation process at ligament region during the test. Crack initiated from notch tip (a), crack growth away from notch tip (b), two crack tip fully coalesced (c), and elongation and gradually fracture of surface layer (d,e). The image was taken from a specimen of 5 wt.% zeolite content with ℓ = 15 mm. White arrows indicate the process stage. White bar length: 2.5 mm.

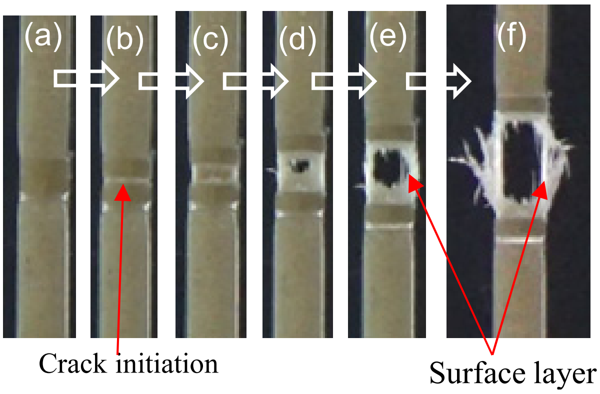

Figure 5. Side view of DENT showing deformation and fracture process, which was taken from a specimen of 5 wt.% zeolite content with ℓ = 13.5 mm.

For polymer-based materials include thermoplastic matrix composites like zeolite-HDPE composite, the plastic zone shape is flat so γ is considerably smaller than 1. Accordingly, the strain rate in the ligament region of zeolite-HDPE has to be considerably greater than that in the metal-based materials when treated with to the same treatment situations, enable the former to reach the strain for development of neck prior to the front of crack reach the ligament center. Consequently, the middle section of the ligament might go by way of the neck development stage prior to the specimen totally fractured.

Change in thickness on ligament section was observed subsequent to the test, and also can be observed during the test. Typical thickness change, as shown in Fig. 5, show that at the neck initiation phase (Fig. 5b) the ligament thickness reduced with the crack growth. Furthermore, at the phase of neck propagation (Fig. 5c), i.e., after the neck has been started through the whole ligament region, the ligament strain in thickness only slightly increased with crack development. The thickness of the ligament is nearly constant of around the initial thickness of 4 mm. The strain is considered to be zero if its direction is parallel to the ligament length, as recommended in the previous study [24]. The strain along the loading direction was found as elongation (see Table 1) in which the elongation decreased with zeolite content increase.

Top, front, and side view of deformation entire ligament region after fracture is presented in Fig. 6, which was taken from 5 wt.% zeolite-HDPE specimen with ℓ = 12 mm. Top and side view presented in Fig. 6a and 6c depict the surface layer that extends at the edge of the FPZ in the ligament section. Front and side view also show that specimens had brittle fracture mechanism in the mid-thickness region, with a small-scale entan–glement of plastic deformation.

The deformation behavior of specimens observed clearly show that critical level of elongation, despite being a general fracture criterion for a ductile specimen like zeolite-HDPE composite, is not acceptable for the fracture process zone in the zeolite-HDPE composite. Another specification for the crack development has been proposed by Chen et al. [31] which was derived from the crack-tip opening angle (CTOA). The typical load-displacement response of tested specimens did not express any presence of a neck development phase, hence it was required re-evaluation of CTOA appro–priateness as a criterion in this work. The rationalization of crack tip angle measurement is expressed in Fig. 7.

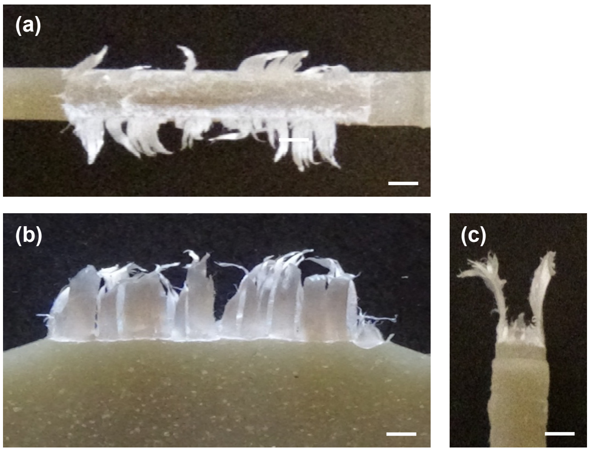

Figure 6. Top, front, and side views of fractured DENT specimen, which was taken from a specimen of 5 wt.% zeolite content with ℓ = 12 mm.

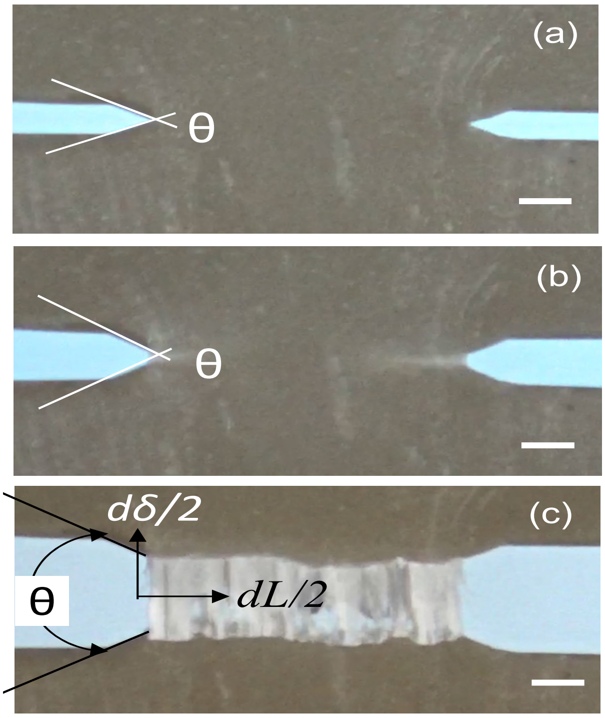

Figure 7. Crack tip angle (θ) of a crack development n DENT test: (a) θ in the neck inception stage, (b) θ in the crack initiation, (c) relationship of θ with elongation δ and ligament length ℓ . Length of white bar correponds to 2,3 mm.

In situ measurement of crack tip angle in the DENT specimen test were rationalized by a though that the relationship between the crack angle (θ), elongation of the specimen (δ), and ligament length (ℓ), as shown in Fig. 7c can be expressed by the mathematical equation as follow [14]:

$$\frac{d\delta}{d\ell} = -\tan\left(\frac{\theta}{2}\right) \qquad (4)$$

To obtain a constant θ value, the $d\delta/d\ell$ parameter must be kept constant during the test. Therefore, DENT specimen testing should be performed at a constant crosshead speed ($d\delta/dt$) and the crack growth rate ($d\ell/dt$) is also kept constant during the test.

4. CONCLUSIONS

A linear relationship was confirmed between the zeolite content in composites and deformation involved such as yielding and necking, a plastic deformation form associated with ductile materials. The crack development in DENT test of zeolite-HDPE annealed composites was similar. The plastic deformation in DENT specimen of zeolite-HDPE composites did not create sufficient strain hardening to keep the development process remain stable. The crack growth is accompanied by the enlargement of the FPZ size met each other at the middle section of the specimen. The difference in the deformation behavior between the neck initiation and neck propagation stages in the DENT specimens tested did not appear clearly. Therefore, the fracture toughness of zeolite-HDPE determined from the test and evaluated using the energy partitioning concept to characterized the fracture stages occurred is appropriate.

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РАЗВОЈ ЛОМА И ПОНАШАЊЕ ДЕФОРМАЦИЈЕ КОД КАЉЕНИХ КОМПОЗИТА СА ЗЕОЛИТ ПУНИЛОМ HDPE У РАВНОМ СТАЊУ НАПОНА

Пурномо, М. Субри, П. Сетјарини

Рад испитује композите са зеолит пунилом HDPE каљене на 90^0C како би се истражио раст прслине и понашање деформације у условима оптерећења на затезање до потпуног лома узорка. Раст прслине и деформација добијени су DENT тестом. У свакој фази развоја лома вредност енергије лома је одређена на основу концепта партиционисања енергије. Процена деформације је вршена у свакој фази процеса лома, како на почетку стварања прслине тако и приликом њеног ширења. Резултати показују да нема разлике у расту прслине, али да деформација варира са варирањем вредности рада лома у свакој фази раста прслине.