

Adiabatic effects on the temperature and rate dependency of the fracture toughness of an ethylene-fluoroethylene film

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ABSTRACT

In this work, the essential work of fracture (EWF) test has been applied to determine the temperature and strain-rate dependency of the fracture toughness of ethylene-fluoroethylene (ETFE) thin films.

EWF tests were conducted at different temperatures (23, 50 and 80 °C) and different cross-head rates (1, 10, and 100 mm/min). At 23 °C, the specific essential work of fracture (w_e) values slightly increase as the strain rate increases, while at 50 °C and 80 °C w_e values decrease. This trend is associated to an increase of the specific essential work for crack propagation ($w_{e,prop}$) while the specific essential work for crack initiation ($w_{e,init}$) decreases with strain rate with a magnitude related to the testing temperature. Thermal observations with an infrared camera revealed an increase of the temperature at the crack tip for the highest strain rate, thus suggesting, for the sample tested at the lowest temperature, the concurrent effects of strain-rate embrittlement and adiabatic heating. Digital image correlation (DIC) analysis revealed an ovoid shape of the outer plastic zone (OPZ) prior to the start of the crack propagation. DIC also indicated that the extension of the OPZ increases linearly with the square of the ligament length.

1. Introduction

As technological progress over recent decades has improved their properties, some plastics have become established as building materials. One of the most interesting among the plastic materials available for application in building constructions is a fluorine-based polymer known as ethylene tetrafluoroethylene (ETFE). ETFE was originally developed in the 1970s by DuPont as a lightweight, heat resistant film to be used in the aerospace industry. Since that time, the film has been used sporadically in various agricultural and architectural applications, such as coverings for greenhouses and protection for solar cells. Brought into the public consciousness thanks to its use as the encapsulating membrane of the Eden Project in Cornwall, UK, a natural evolution of Buckminster Fuller's Biosphere concept in 2001, architects are now exploiting ETFE's capabilities to express new aesthetic and replace costlier transparent and translucent materials.

ETFE has very good mechanical properties, excellent chemical resistance, electrical characteristics, outstanding resistance to weather and aging make it suitable in a large spectrum of applications [1]. The main use of ETFE in building is as a cladding material since it allows the construction of spectacular architectures with unusual forms. Furthermore, due to the fluoro-groups in the molecular chains, ETFE is shatterproof and self-cleaning [2]. Its main advantages over glass are its lower weight, higher light transmission, and its improved resilience. The performance of ETFE in architectural application is better than glass due to the efficient

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Nomenclature		w_p	specific non-essential work of fracture
		$w_{e,prop}$	specific essential work for crack propagation
Symbols		$w_{e,init}$	specific essential work for crack initiation
β	plastic zone shape factor	Acronyms	
ΔH_m	melting enthalpy	CCD	Couple Charged Device
ΔH_r	melting enthalpy of 100% crystalline polymer	DIC	Digital Image Correlation
σ_y	yield stress	DMTA	Dynamic Mechanical Thermal Analysis
E_y^*	activation energy	DSC	Differential Scanning Calorimetry
L	ligament length of the DENT specimens	E_y^*	Activation Energy
K	Boltzmann's constant	ESIS	European Structural Integrity Society
t	thickness of the DENT specimens	ETFE	Ethylene TetraFluoroEthylene
T	Kelvin temperature	EPF	Essential Work of Fracture
V^*	activation volume	FPZ	Fracture Process Zone
W_f	total work of fracture	ISO	International Organization for Standardization
w_f	specific total work of fracture	OPZ	Outer Plastic Zone
W_e	essential work of fracture	TGA	Thermogravimetric Analysis
w_e	specific essential work of fracture		
W_p	non-essential work of fracture		

thermal insulation and good noise absorption, especially when used in multilayers [3]. In particular, the lightness and the good mechanical properties of ETFE permit the construction of active and adaptable buildings containing movable parts and transformable elements that are able to react instantaneously to changes of the environment or the user demand, such as cushion structures [4]. These factors are becoming more and more important as the demand for green building with superior energy efficiency is increasing [5,6].

The fracture toughness is a very important property for construction materials since the presence of flaws is not completely avoidable in the fabrication or service of a component. The Essential Work of Fracture (EPF) approach is one of the few experimental methods to evaluate the fracture toughness of highly ductile materials under plane-stress conditions (thin films or sheets) [7]. The EPF approach is based on the separation of the work expended in the fracture process zone (FPZ), which is considered to be essential for the formation of the fracture surfaces, from other dissipative contributions to the total energy (such as plastic deformation) dissipated in the outer plastic zone (OPZ) [8–10].

In this study, the essential work of fracture approach has been adopted to investigate the plane-stress fracture behavior of a commercial ETFE thin film. The aim of the work is to analyze the effects of temperature and strain rate on the fracture parameters. In addition, a partitioning of the essential and non-essential work of fracture into yielding and necking/tearing related terms was performed and the effect of adiabatic heating was considered. The shape of the outer plastic zone was evaluated using Digital Image Correlation (DIC).

2. Materials and methods

2.1. Materials

The material investigated in the present study is a commercial grade of ethylene-tetrafluoroethylene block copolymer (ETFE) provided by PATI S.p.A. (San Zenone Degli Ezzelini, Treviso-Italy). It is an extruded transparent sheet with an average thickness of 200 μm , provided in A4 size (210 \times 293 mm) samples. According to the technical datasheet from the supplier, the film is characterized by a light transmittance of 89% (EN 2155-5). The films are also characterized by good chemical resistance especially against acid rain and aggressive substances which makes it a preferable material for outdoor civil applications.

2.2. Characterization techniques

2.2.1. Physicochemical characterization

The thermal stability of the material was determined by thermogravimetric analysis (TGA) by a TA Instrument Q5000 at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ under a nitrogen flow of 50 ml/min from room temperature to 700 $^{\circ}\text{C}$. The temperature associated with 5% sample mass loss and the residual mass at 700 $^{\circ}\text{C}$ were determined.

Differential scanning calorimetry (DSC) measurements were conducted by a Mettler DSC 30 device. The experiment was operated at constant heating rate of 10 $^{\circ}\text{C}/\text{min}$ in nitrogen atmosphere on 5 mg sample of ETFE film, in a temperature range of -170 to 300 $^{\circ}\text{C}$. The specific heat of melting and crystallization were calculated from the area under the endo- or exothermal peaks, respectively. The degree of crystallinity was calculated using Eq. (1):

$$\Delta X_c = \frac{\Delta H_m}{\Delta H_r} \times 100 \quad (1)$$

where ΔH_m is the melting enthalpy of ETFE film which corresponds to the area under the melting peak. ΔH_f , that represents the melting enthalpy of 100% crystalline ETFE polymer, has been taken equal to 113.4 J/g [11].

Dynamic Mechanical Thermal Analysis (DMTA) tests were performed on ETFE film by a TA Instrument DMA-Q800 device under tensile mode on rectangular strips of 5 mm in width and 10 mm in length. The tests were carried out at a frequency of 1 Hz, from -150°C to 150°C at a heating rate of $3^\circ\text{C}/\text{min}$. The storage modulus (E'), the loss modulus (E'') and loss tangents ($\tan \delta$) were obtained as a function of temperature.

The elastic modulus of ETFE film was measured with the same TA Instrument DMA Q800. The test was performed in tensile mode applying a constant deformation rate of $200\ \mu\text{m}/\text{min}$ at temperatures of 23°C , 50°C and 80°C .

Uniaxial tensile tests at high strain levels were also performed to evaluate the effect of testing speed and temperature on the yield behavior of ETFE film. In particular, an Instron 4502 electromechanical testing machine equipped with a 100 N load cell was used. ISO 527 1BA specimens were cut from ETFE film and tested at different crosshead speeds (1, 10 and 100 mm/min) and temperatures (23, 50 and 80°C).

2.2.2. Fracture toughness characterization

The main concepts regarding the essential work of fracture approach are summarized in [Appendix A](#).

The double edge notched tension (DENT) specimens for the EWF test were prepared according to the ESIS-TC4 protocol [12]. Notches were sliced in ETFE strips with a width of 30 mm with the aid of a scalpel blade to generate ligament lengths of 7, 9, 11, 13 and 15 mm. The initial distance between grips was fixed at 50 mm. At least 5 specimens were tested for each ligament length. EWF tests were performed with an Instron 4502 electromechanical testing machine equipped with a 1 kN load cell and a thermostatic chamber.

Although the EWF approach has not been standardized, testing protocols have been developed by ESIS TC4 committee and proposed to ensure the reliability of this approach [7]. To achieve plane-stress conditions, the ligament length must be not less than three to five times the film thickness. At the same time, the ligament length must be not greater than two times the plastic zone radius or one third of the specimen width to avoid edge effects. To apply the EWF approach, a complete yielding of the ligament must precede the onset of crack initiation. To ensure that the fracture has occurred after the full ligament yielding, a stress criterion has been introduced, which is based on the maximum net section stress (σ_{\max}) obtained dividing the maximum peak load by the ligament cross section. σ_{\max} should be independent of ligament length. For all the tested specimens, an average value of σ_{\max} , denoted as σ_m , was determined, and the specimens for which $\sigma_{\max} < 0.9 \sigma_m$ or $\sigma_{\max} > 1.1 \sigma_m$ were rejected. The total work of fracture (W_f) was calculated for each valid specimen from the area under the load–displacement curve, and the specific work of fracture (w_f) was plotted against the ligament length. A least square regression line was used to fit the data, and the points lying outside more than two times the standard deviation range from the best-fit line were eliminated. Having rejected these points, a final least square regression line was obtained considering the remaining data to give the intercept, which is called the essential specific work of fracture (w_e), and its slope, which is called the non-essential specific work of fracture.

A FLIR/E63900 infrared camera was used to observe the effect of adiabatic heating ahead of the crack tip for EWF tests performed on DENT specimen of ETFE. Moreover, all experiments were recorded with a digital camera synchronized with the load-displacement acquisition system in order to monitor the crack propagation.

2.2.3. Digital image correlation method

The digital image correlation (DIC) method is an optical approach employing tracking and image registration techniques for tracking speckle movements in digital images [13]. In this paper DIC is used to provide deformation and strain measurements on the ETFE films. In this method, optical measurements were performed using a 3D DIC system (Q-400; Dantec Dynamics GmbH, Ulm, Germany and Denmark). A speckle pattern using black spray paint is used to create the targets on the specimen. A resolution of 5 megapixels is used for the couple charged device (CCD) cameras together with a 35 mm lens on each camera. The output signal from the loading system is synchronized with the image correlation system so that each frame is correlated to the actual loads applied to

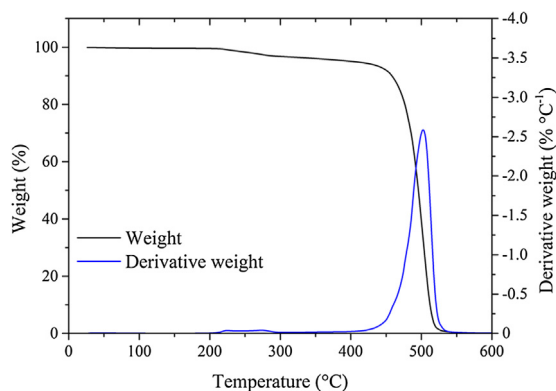


Fig. 1. Thermogravimetric curve of the investigated ETFE film and its derivative.

the specimen at that point.

3. Results and discussion

3.1. Physicochemical characterization

Thermogravimetric analysis (TGA) was used to observe the thermal resistance of the ETFE film. ETFE is a fluoropolymer which is characterized by high thermal stability. However, it exhibits a small amount of degradation at processing temperature and it undergoes decomposition in a single stage. In fact, in Fig. 1 it is possible to observe that the material is characterized by a single stage decomposition starting from 392 °C with a maximum degradation rate at 502 °C as indicated by the peak on the TGA derivative curve.

A differential scanning calorimetry curve of ETFE is presented in Fig. 2. A sharp endothermic peak can be observed in the heating curve at 271 °C, that corresponds to the melting temperature of the crystalline regions. The degree of crystallinity, calculated according to Eq. (1), is 43.5 wt%.

The evolution of the tensile storage modulus (E'), loss modulus (E'') and loss factor ($\tan \delta$) with temperature is reported in Fig. 3. The storage modulus is quite stable in a wide range around ambient temperature. This behavior is positive for building application due to a consistency in its elastic response around the in-service temperatures. The peak in both the loss modulus and $\tan \delta$ curves at -40 °C represents the glass transition temperature of this material [1]. The $\tan \delta$ curve reveals another molecular transition at 120 °C which represents the relaxation of the alpha phase [1]. The peak on the loss modulus at 90 °C, that appear as a little shoulder on the previous peak of the $\tan \delta$, is associated with the crystal-crystal transition from an orthorhombic to a hexagonal molecular structure [14].

Tensile test results obtained with the TA Instrument DMA Q800 are summarized in Table 1. The elastic modulus decreases with the temperature, in line with DMA complex modulus results.

Fig. 4 shows the yield stress values obtained from the uniaxial tensile test. The yield stress decreases strongly with temperature, while increasing cross-head speed results in only a slight increase.

Bauwens-Crowet et al. [15] reported that tensile yield stress data for PVC depended on temperature and strain rate according to Eyring theory [16,17] as expressed in the simplified form [18]:

$$\frac{\sigma_y}{T} = \frac{E_y^*}{V^*T} + \frac{k}{V^*} \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0} \quad (2)$$

where E_y^* is an activation energy, V^* is an activation volume, T is the temperature in Kelvin degrees and k is the Boltzmann's constant. Plotted in Fig. 5 according to Eq. (2), our ETFE results show the activation energy decreasing slightly and the activation volume decreasing remarkably from $2.85 \times 10^{-3} \text{ m}^3 \text{ mol}^{-1}$ at 23 °C to $5.03 \times 10^{-3} \text{ m}^3 \text{ mol}^{-1}$ at 80 °C. The notable increase in the activation volume could help to explain the higher relation between the yield stress and the temperature due to the fact that the role of the temperature is played by the free volume as reported in literature [19].

3.2. Fracture behaviour

3.2.1. EWF parameters

Load-displacement curves from EWF tests were similar for a wide range of ligament lengths, temperatures and cross-head speeds. This indicates that the fracture mechanism is independent of the ligament length, this being one of the basic requirements for the EWF approach. Results at different cross-head speeds for a ligament length of 13 mm are shown in Fig. 6. The shape of the load-displacement curves at different ligament length, different temperature and different cross-head speeds are similar to one another for every specimen. The self-similarity in the load-displacement curves indicates that fracture mechanism is independent from the

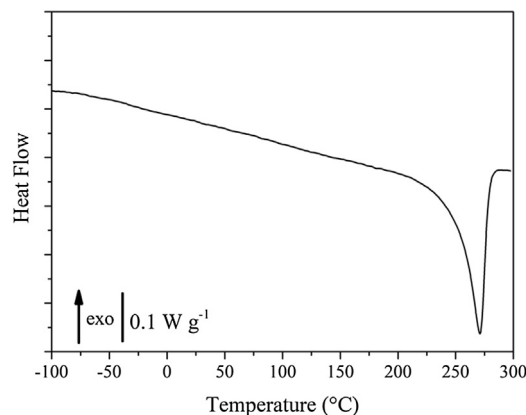


Fig. 2. Differential scanning calorimetry curve for ETFE film.

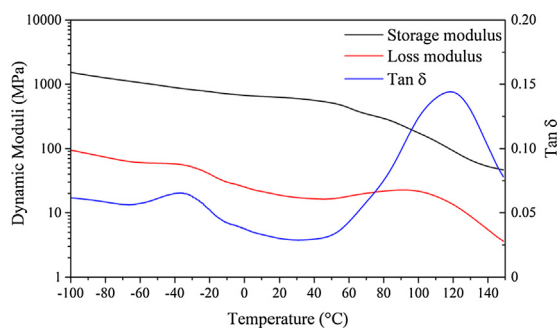


Fig. 3. Storage, loss modulus and tan delta curves for ETFE in a range of temperature from -100°C to 150°C .

Table 1

Elastic modulus and storage modulus at various temperatures.

Temperature ($^{\circ}\text{C}$)	Elastic modulus (MPa)	Storage modulus (MPa)
23	987 ± 6	619 ± 1
50	576 ± 16	505 ± 5
80	396 ± 2	290 ± 2

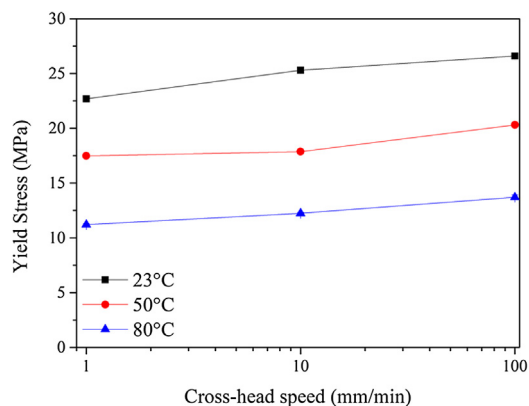


Fig. 4. Yield stress for different temperatures as function of the crosshead speed.

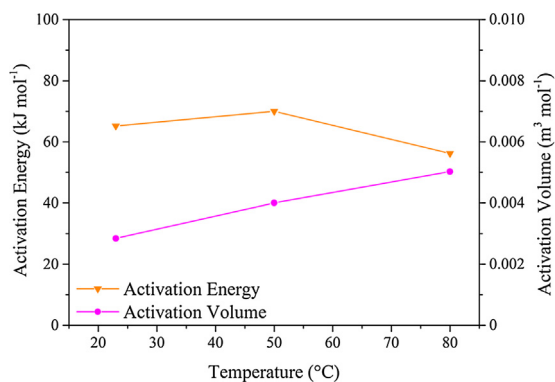


Fig. 5. Parameters of Eq. (2).

ligament length which is one of the basic requirements for the application of the EWF approach. This shape of load-displacement curves is peculiar to the investigated ETFE film, even if similar trends were found in other polymeric materials [10,20,21]. Like the yield stress, the maximum load increases slightly with increasing testing speed and decreases with increasing temperature. All the specimens displayed a large plastic deformation zone surrounding crack tip and preceding the crack growth.

The area under the resulting load-displacement curves was integrated to provide the total work of fracture value (W_f) of the

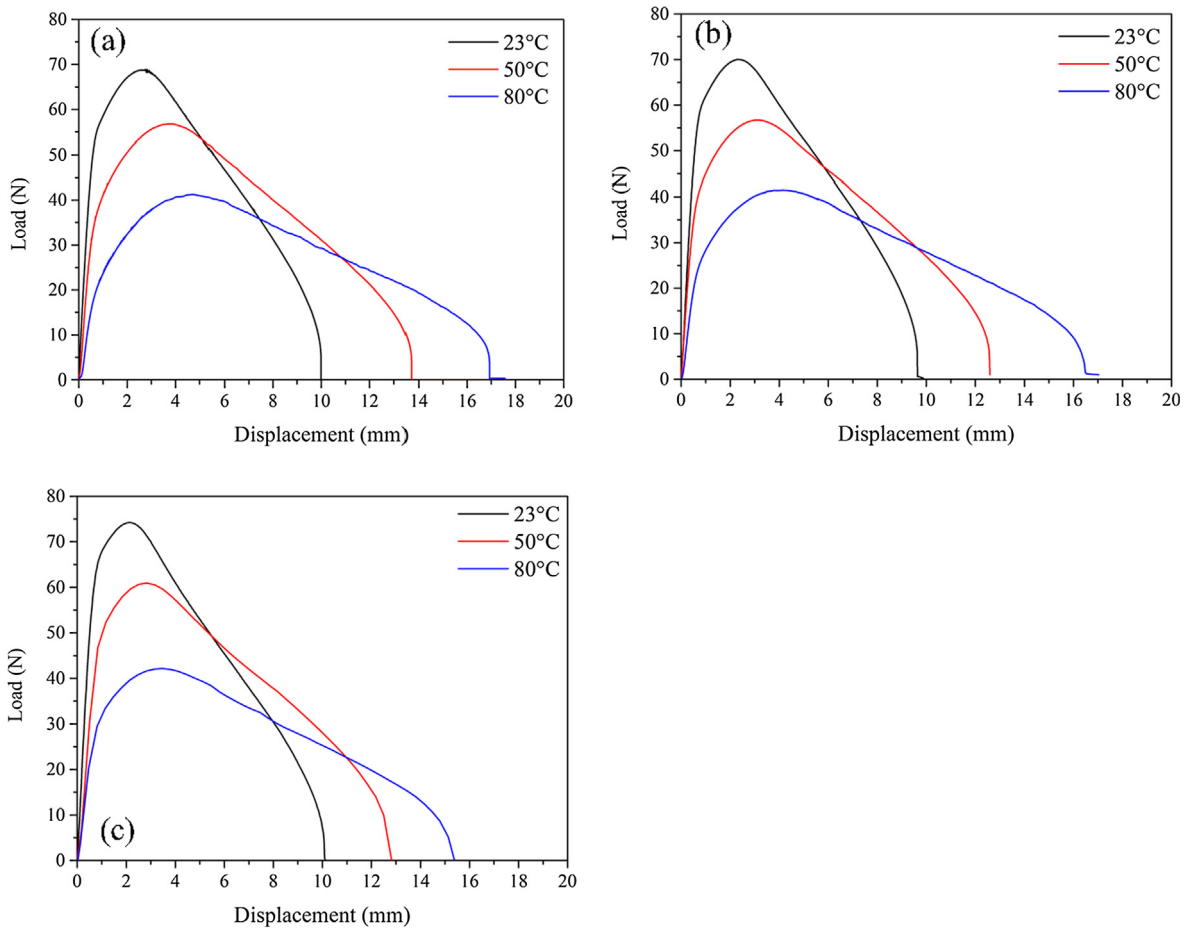


Fig. 6. EWF curves for 13 mm ligament length DENT specimens tested at various cross-head speeds: (a) 1 mm/min, (b) 10 mm/min and (c) 100 mm/min.

corresponding sample. Plots of the specific total work of fracture (w_f) as a function of the ligament length were constructed and fitted by linear regression to obtain w_e and βw_p parameters as functions of crosshead speed (Fig. 7). A graphical representation of the trend of parameters as a function of the cross-head speed is reported in Fig. 7. The essential work of fracture parameter increases as the temperature increases for low deformation speed, while at the highest cross-head speed of 100 mm/min the highest value of w_e is found for the test at room temperature. The decrease of the essential work of fracture (i.e. lower toughness) with the increase of

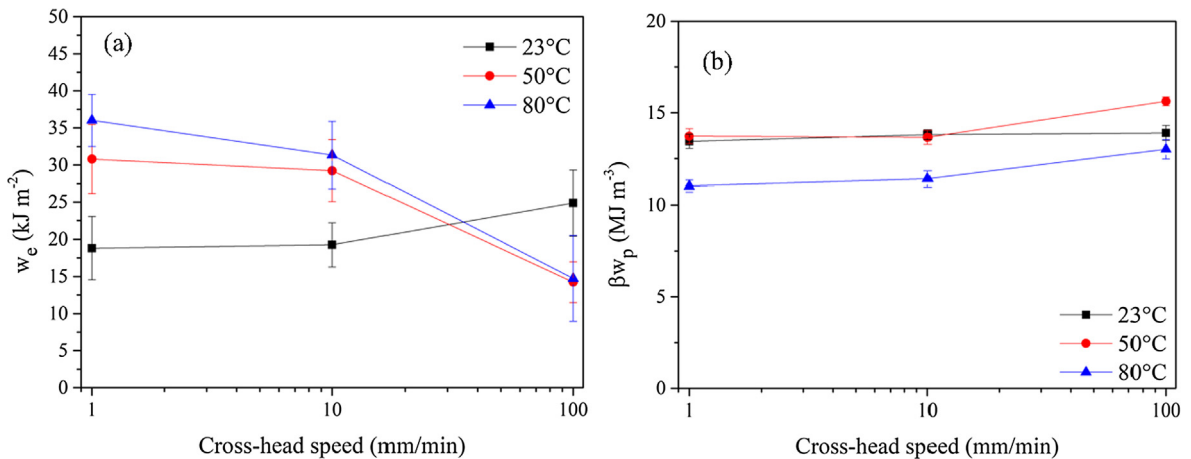


Fig. 7. Essential (a) and non-essential (b) work of fracture at different testing temperatures against crosshead speed.

deformation rate is caused by the reduction of the molecular motion, known as strain rate embrittlement [22]. EWF approach clearly showed that the energy dissipated during the fracture of DENT specimen is lower for the test at 80 °C where, according with the Eyring model, the activation volume is higher, so the molecules can move more easily and the activation energy for their movements is lower.

The total specific energy spent for the fracture of DENT specimen could be partitioned in two terms: a specific work for crack initiation (w_{init}) and a specific work for crack propagation (w_{prop}) [23,24]. Therefore, Eq. (A4) could be rewritten as:

$$w_f = w_{init} + w_{prop} = w_e + \beta w_p L \quad (3)$$

The energy spent for crack initiation and propagation can be plotted against the ligament length and following the same procedure adopted for w_f :

$$w_{init} = w_{e,init} + \beta' w_{p,init} L \quad (4)$$

$$w_{prop} = w_{e,prop} + \beta' w_{p,prop} L \quad (5)$$

The specific essential work of fracture can therefore be written as:

$$w_e = w_{e,init} + w_{e,prop} \quad (6)$$

As documented in Fig. 8 for a couple of representative cases, video records showed that cracks start to propagate slightly before the maximum load was reached.

Therefore, on the basis of the video-records, this crack-onset point was determined for each test and used to divide the area under the load-displacement curve in two parts: one from the start of the experiment until crack initiation ($w_{e,init}$) and another up to complete fracture ($w_{e,prop}$). Fig. 9 plots $w_{e,init}$ and $w_{e,prop}$ as function of the cross-head speed at 23, 50 and 80 °C. The initiation-related essential work of fracture parameter ($w_{e,init}$) was found to decrease with the increasing of the testing speed for test at 50 °C and 23 °C where it reached values close to zero.

The specific essential work for crack propagation dominates in determining the specific essential work of fracture (w_e) and it has a similar trend. As previously reported, the decreasing in $w_{e,prop}$ could be explained by a strain embrittlement effect. The opposite trend for test at room temperature could be explained by adiabatic heating in the ligament region [22], which can increase the molecular mobility and hence the toughness.

Strain embrittlement and adiabatic heating compete against each other so the relation of the fracture parameters with the strain rate is non-linear. In fact, adiabatic heating was directly observed through a thermo-camera for the specimen at room temperature at the highest deformation rate. In fact, as documented in Fig. 10a, it is possible to observe a strong temperature increase ahead the crack tip for the test at 100 mm/min and 23 °C (Fig. 10b). At the same time, no increase of temperature was detected for the tests performed at higher temperature.

3.2.2. Plastic zone shape factor evaluation

The parameter β (shape factor) is proportional to the area of the yielded region at the crack tip. Its determination can be interesting in some cases, such as for the calculation of J-integral. The ESIS-TC4 protocol proposes some typical plastic zone shape and their relative β value given the length and the height of the plastic zone [12]. Other authors proposed different approaches for the calculation of β such as a combination of elliptical shapes [25]. In our approach, a real time video of the strain in the specimen was acquired through DIC and the area of the yielded zone was precisely measured (Fig. 11). For extremely large strains after the maximum load, the speckle pattern becomes sufficiently distorted in the high strain region, but the strain outside the intense deformation zone is accurately captured.

The plastically deformed area plotted against the square of the ligament length gives rise of the β value as the slope of the least-

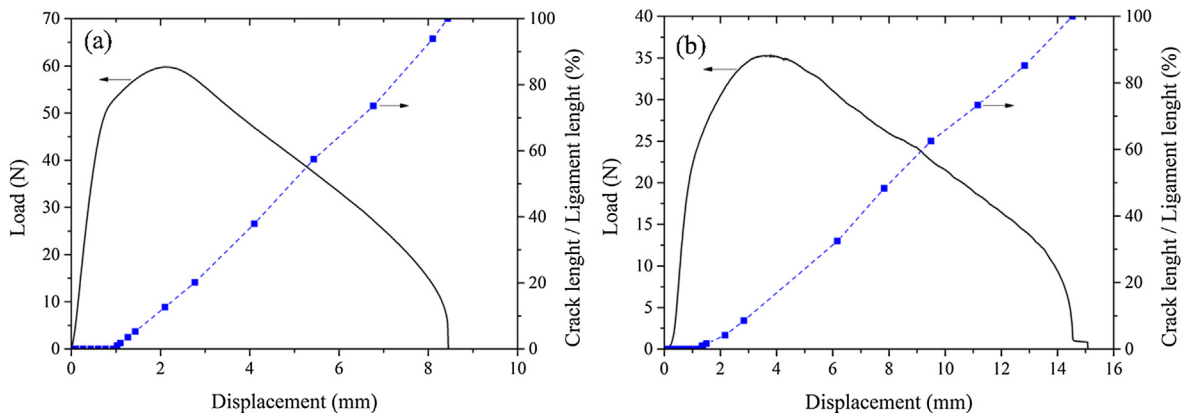


Fig. 8. Load–displacement curve and evolution of the crack length during the fracture tests of ETFE specimen tested at 23 °C (a) and 80 °C (b) at a cross-head speed of 10 mm/min.

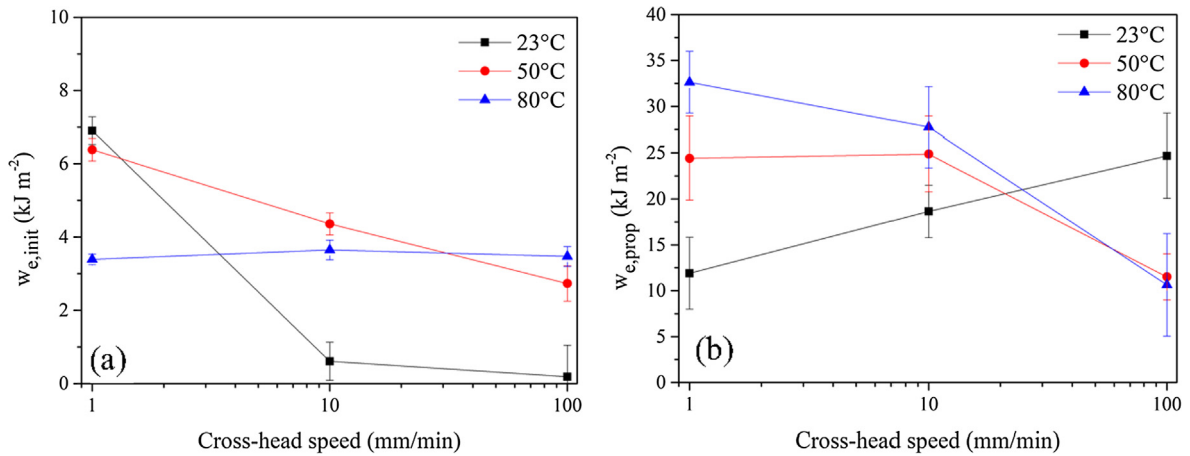


Fig. 9. Specific essential work of fracture for crack initiation (a) and for crack propagation (b) at different temperature against cross head speed.

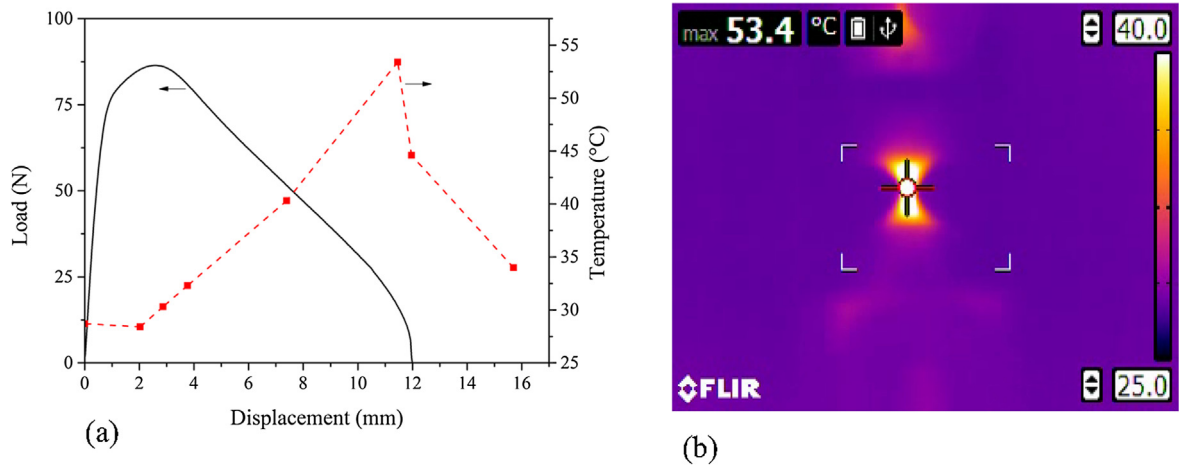


Fig. 10. (a) Load and temperature at the crack tip against displacement and (b) infrared picture of the specimen during a test at 100 mm/min and 23 $^{\circ}\text{C}$.

squares regression line used to fit the values. For the sample tested at 10 mm/min and room temperature, the plastically deformed area appeared as an oval with a shape factor results to be 2.63, thus, with a height equals to 3.35 times the ligament length (Fig. 12).

4. Conclusions

Both temperature and deformation rate affect the fracture toughness of ETFE films. In particular, two competing phenomena determine the specific essential work of fracture values: adiabatic heating and strain rate embrittlement. The rate-dependency of the specific essential work of fracture displayed different trends at different temperatures. At higher temperature, w_e decreased due to strain embrittlement effect while at room temperature w_e increased as the strain rate increased. Infrared images showed a rise in temperature near the crack tip that could increase the molecular chain mobility, and the fracture toughness. The yielding phenomenon at different temperature and different strain rate, was analyzed by the Eyring model. A temperature-induced increase of the activation volume and a decrease in the activation energy for the plastic flow of polymer macromolecules was observed. These results well agree with the decreasing trend of βw_p as the temperature increases. The evolution of the plastic zone around the ligament length was evaluated with a DIC method. This approach could be helpful to obtain precise values of the shape factor β .

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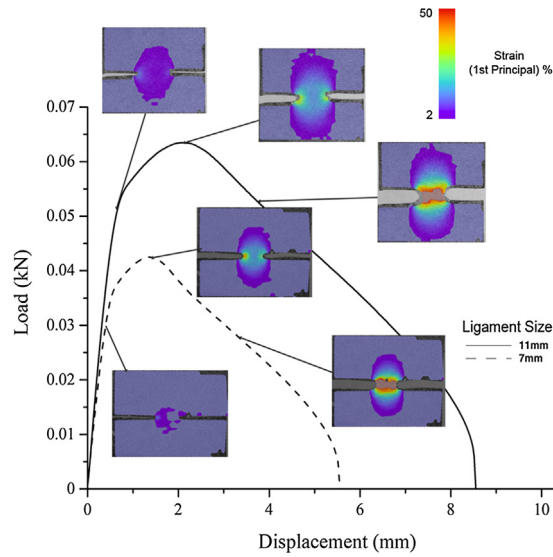


Fig. 11. Progression of area of plastic zone and crack growth during loading of ETFE specimens as detected by DIC measurements on samples tested at 23 °C and 10 mm/min.

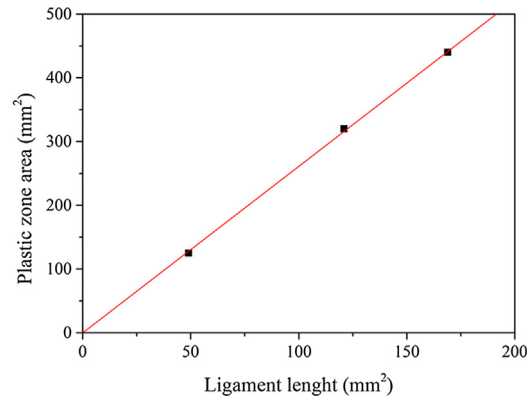


Fig. 12. Area of the plastic zone vs the square of the ligament length for the test at 10 mm/min and 23 °C as determined from DIC.

Appendix A

According to the EWF approach, the total energy consumed in the planar fracture process of the double edge notched tension (DENT) specimen could be divided in two terms, the essential work of fracture (W_e), that represents the energy consumed in the fracture process zone, and the non-essential work of fracture (W_p), that stands for the energy dissipated in the outer plastic volume in which a number of energy dissipation mechanisms may occur. Therefore, the total work of fracture (W_f) calculated as the integral of the load-displacement curve could be expressed by:

$$W_f = W_e + W_p \quad (A1)$$

Considering W_e proportional to the ligament area and W_p proportional to the volume of the outer plastic region, the following specific terms could be written:

$$w_e = W_e/(tL) \quad (A2)$$

$$\beta w_p = W_p/(tL^2) \quad (A3)$$

where w_e is the specific essential work of fracture, w_p is the non-essential work of fracture, t is the thickness of the specimen, L is the ligament length and β is a plastic zone shape factor. Combining Eqs. (A1)–(A3) it is possible to obtain:

$$w_f = W_f/(tL) = w_e + \beta w_p L \quad (A4)$$

where w_f is the specific work of fracture, calculated as the total work of fracture divided by the ligament length area.

Appendix B. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.engfracmech.2019.03.005>.

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