

Fracture toughness of rubber in quasi-static conditions: how to experimentally obtain results intrinsic to the material?

O. Popovych, S. Agnelli

University of Brescia, Brescia, Italy

C. Marano

Politecnico di Milano, Milan, Italy

B. Schrittester

Scioflex GmbH, Wien, Austria

W. Balasooriya

Polymer Competence Center Leoben GmbH, Leoben, Austria

ABSTRACT: Fracture toughness, defined as the energy dissipated to advance a crack by a unit area, is an intrinsic material property, which should not depend on the geometry and dimensions of the specimen used to obtain it. One of the most frequently used test configurations to determine rubber fracture toughness at crack initiation in quasi-static conditions is the single edge notched in tension specimen (SENT). The aim of this work is to quantitatively define the limitations of specimen dimensions needed to obtain an intrinsic value of fracture toughness for a nitrile-butadiene rubber (NBR), and to investigate the possible effects of the test procedure on the results. These show that a minimum width of 10 mm and a minimum height/width ratio of 4.6 are necessary to obtain a fracture toughness independent of specimen dimensions.

1 INTRODUCTION

Rubber is an irreplaceable material for both mainstream engineering applications (e.g.: tires, sealing and dampers) and for advanced applications in the fields of soft robotics, biomedicine and stretchable electronics, for example. For these applications, the proper characterization of fracture resistance is useful both for the development of tougher materials, and to guarantee structural integrity of components.

The first and most used approach to describe the fracture behaviour of rubber at the macro-scale was proposed by Rivlin (Rivlin and Thomas 1953) and is based on the energy release rate concept. Fracture toughness of rubber is defined as the energy dissipated to advance a crack by a unit area. In principle, it is an intrinsic material property, which should not depend on the geometry and dimensions of the specimen used to obtain it. Based on the fundamental work of Rivlin and Thomas, much research has been done to develop experimental methods to measure rubber fracture toughness considering different test pieces, calculation approaches, and loading conditions.

One of the most frequently used test configurations to determine fracture toughness at initiation in quasi-static conditions is the SENT specimen (see Figure 1a). The specimen is a strip with a lateral notch loaded in tension. Several research groups used this test method, but no systematic work has been done yet on the limitations of specimen dimensions needed to obtain a reliable value of fracture toughness, and on the effects of the test procedure on the results. The aim of this work is to draw guidelines useful for the

obtainment of an intrinsic value of fracture toughness at crack initiation for rubber.

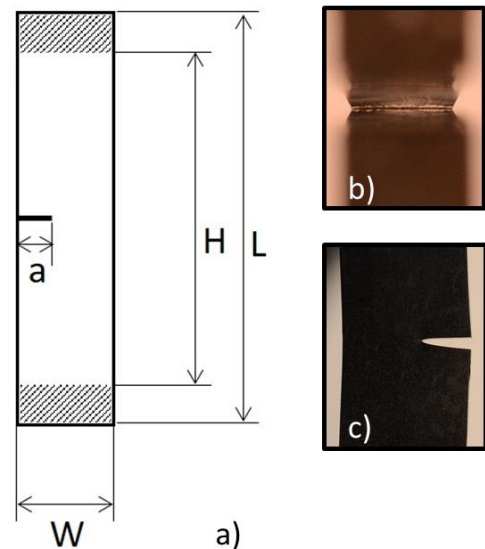


Figure 1. Schematic representation of SENT specimen geometry (a); frames taken during a test on an SENT specimen reproducing the front view (b) and lateral view (c) of the crack tip.

An NBR is used for this study, SENT specimens with different dimensions are tested and fracture toughness is evaluated in quasi-static conditions by the tearing energy parameter. The effects of specimen width, length and size are investigated. Moreover, various techniques for specimen notching and for monitoring fracture initiation are used, to evaluate their effects on the results.

2 EXPERIMENTAL DETAILS

2.1 Test procedure

The SENT specimen is stretched in a tensile test machine at a constant displacement rate. During the test the load and the crosshead displacement are recorded. At the same time an optical system is used to monitor fracture onset. This can be observed either with a front view or with a lateral view. In the front view the optical device is pointed at the notched side surface of the specimen, and the notched specimen surfaces of black rubber are previously covered by a white powder (e.g. talc). From the images taken during the test (see Figure 1b) the new surfaces are clearly visible as fracture initiates, due to the color contrast between the white sprayed powder and the black, newly formed, surface of the rubber. The distance between the edges of the sharpened notch tip during the fracture process (CTOD*) can thus be measured, and fracture onset is conventionally defined as the time corresponding to a CTOD* equal to 0.1 mm, according to a procedure proposed in (Ramorino et al. 2010). In the lateral view, the optical device is pointed at the lateral specimen surface, as much parallel to it as possible (see Figure 1c). The notch tip has initially a rounded shape and then it becomes sharpened. Fracture onset is defined as the time at which the sharp tip forms.

Once the critical point corresponding to fracture onset is identified on the load-displacement curve, fracture toughness at initiation (u_c) is calculated according to the following equation (Agnelli et al. 2020):

$$u_c = (2\pi/\lambda^{0.5}) \cdot a \cdot U_i / (t \cdot W \cdot H) \quad (1)$$

where a , t , W and H are the specimen notch length, thickness, width and height, respectively (see Figure 1a), λ is the stretch ratio (crosshead displacement/ H) at the point of fracture onset, and U_i is the area under the load-displacement curve up to the critical point.

2.2 Material and test conditions

A commercial grade NBR reinforced with carbon black was kindly provided in the form of 2 mm thick sheets by Semperit Technische Produkte GmbH, Austria. Tensile stress at break and tensile strain at break were 19 MPa and 120%, respectively, measured in uniaxial tensile tests by the authors.

SENT specimens were cut from the sheets with various dimensions (see Table 1), and are labelled “HxW”, where H and W are specimen height and width, respectively. For all specimens, $a/W=0.4$, and $t=2$ mm. The “standard” specimen is “100x15”. A minimum of five specimens were tested for each condition.

Table 1: u_c [kJ/m²] values obtained with different experimental conditions (test conditions or specimen dimensions)

TEST CONDITIONS (on 100x15 specimens)		SPECIMEN DIMENSIONS (with front view, Gillette push)	
Front view		Standard	
Gillette push	0.56±0.05	100x15	0.56±0.05
Gillette zig zag	0.58±0.08	Length effect	
Stanley push	0.58±0.16	130x15	0.60±0.03
Stanley zigzag	0.59±0.09	70x15	0.63±0.02
Lateral view		40x15	1.00±0.22
Gillette push	1.27±0.14	10x15	2.77±0.64
		Width effect	
		167x25	0.57±0.06
		100x20	0.60±0.04
		100x10	0.61±0.11
		100x5	0.72±0.09
		100x3	0.89±0.06
		50x2	0.96±0.07
		Size effect	
		67x10	0.66±0.10
		33x5	0.92±0.14

Notches were manually introduced into the samples with different blades and insertion directions. Two types of blade were used: a razor blade (labelled “Gillette”), with a tip radius of 0.3 μ m and a maximum thickness of 0.1 mm, and an industrial blade (labelled “Stanley”), with a tip radius of a few μ m and a maximum thickness of 0.4 mm. Two different modes of blade insertion were adopted: in “push” mode the blade was pushed inside the material from the lateral side, moving it along direction x in Figure 2, in “zig-zag” mode the blade was slid back and forth along direction z , and slightly pushed along direction x .

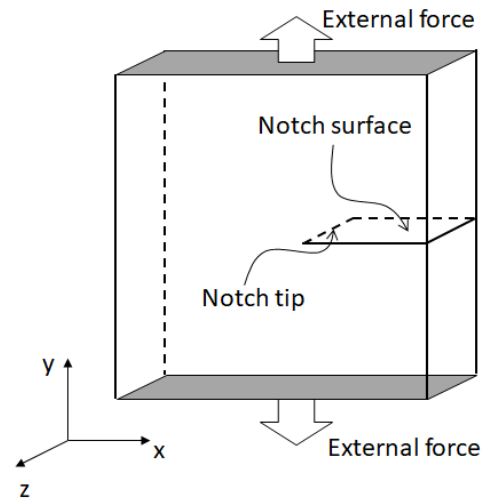


Figure 2. A section of a specimen with indication of notch surface.

Fracture tests were carried out with a Instron Series 3366 test machine, equipped with a 500 N load cell and screw action grips, at 0.1 min⁻¹ of nominal

elongation rate (crosshead rate/H). A digital camera shooting a picture every second was used as optical system to capture images, and image elaboration was performed by ImageJ software.

3 RESULTS AND DISCUSSION

The results of fracture toughness obtained with different experimental conditions and with different specimen dimensions are reported in Table 1.

The results show that the notching methods do not produce significantly different values of u_c , measured with a frontal observation of the notch. Therefore, the method “Gillette push” was selected for all the other investigations since it provides results with the lowest dispersion (standard deviation) and it is the easiest from the practical point of view, i.e. it requires to apply less pressure with hands.

Fracture toughness value of the standard specimen (100x15) is greatly affected by the method of fracture onset monitoring: the front view provides a value ($0.56 \pm 0.05 \text{ kJ/m}^2$) less than half compared to the one obtained with the lateral view ($1.27 \pm 0.14 \text{ kJ/m}^2$). To explain this effect, the authors hypothesize that the two modes of observation actually identify two different physical phenomena: while the frontal view allows the initiation to be identified at the first signs of damage of the notch tip, the lateral observation method seems to be related to a macroscopic variation in blunting, which occurs when the fracture approaches the stage of propagation at a constant rate.

The investigation of the effect of specimen dimensions was therefore carried out with the conditions providing the most conservative value of fracture toughness, i.e. Gillette push and front view.

Length was both increased and decreased with respect to the standard value (100 mm) and u_c results are graphically represented in Figure 3.

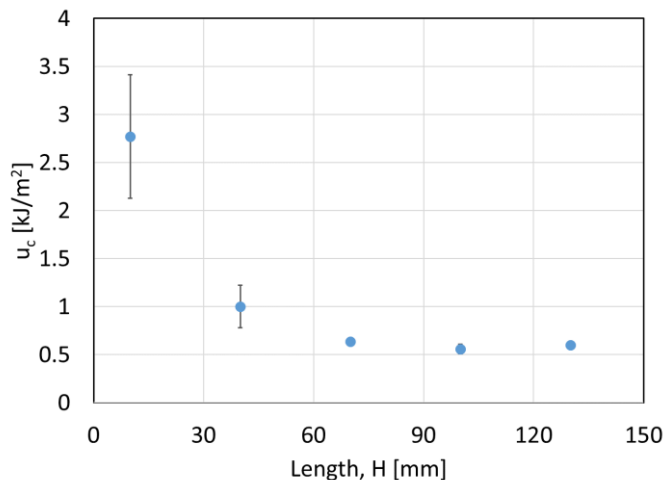


Figure 3. u_c results of SENT specimens with different length (Gillette push and front view) and 15 mm of width.

The data of Table 1 reported in Figure 3 show that u_c holds a stable value for specimen length values

around the standard value, down to 70 mm (corresponding to $H/W=4.7$). Below this value the u_c increases. The main reason to explain the increase of fracture toughness with reducing the length below the standard value, is that the assumptions used to derive Equation 1 are no longer valid. More precisely, the quantity $U_i/(t \cdot W \cdot H)$ in Equation 1 ideally is a measure of the strain energy density (SED) in the bulk of the specimen, i.e. far from the notch. Actually, that quantity is calculated as an average value of SED over all the specimen volume, and is used as a measure of SED far from the notch under the hypothesis that the fracture process zone is small compared to the specimen volume. This hypothesis is true in case of specimens having a high H/W ratio, and a low a/W ratio, as suggested by Lake (Lake et al. 2000). However, when H is not sufficiently long, the higher SED in the fracture process zone is no more negligible, and the average SED is higher than the local SED far from the notch. Besides this reason, when the specimens are stump (10x15) other effects can come into play, such as a partial slippage of specimen out of the grips due to bending, or an overlapping of the region affected by clamping with the fracture process region.

The effect of width was also investigated by keeping the specimen length fixed (100 mm), and the results are plotted in Figure 4. Actually the length was modified in two cases: the specimen 50x2 substituted the 100x2 specimen, since it was problematic to properly cut such a long and thin strip, and the specimen 167x25 substituted the 100x25 specimen, which had a H/W ratio equal to 4, in order to avoid effects due to the reduced L/W ratio, as shown before.

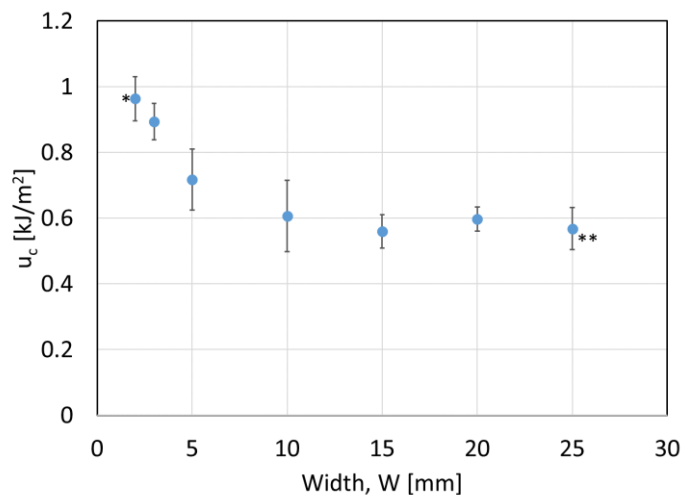


Figure 4. u_c results of SENT specimens with different width (Gillette push and front view) and length of 100 mm (except for: * $H=50$ mm, ** $H=167$ mm).

The results in Figure 4 show that u_c holds a fairly constant value from a width of 25 mm down to 10 mm, whereas below 10 mm width fracture toughness increases, probably due to the interference of the specimen edge with the fracture process zone.

Finally, Figure 5 shows u_c results obtained varying both specimen width and length, but keeping H/W ratio equal to 6.7, as in the standard specimen.

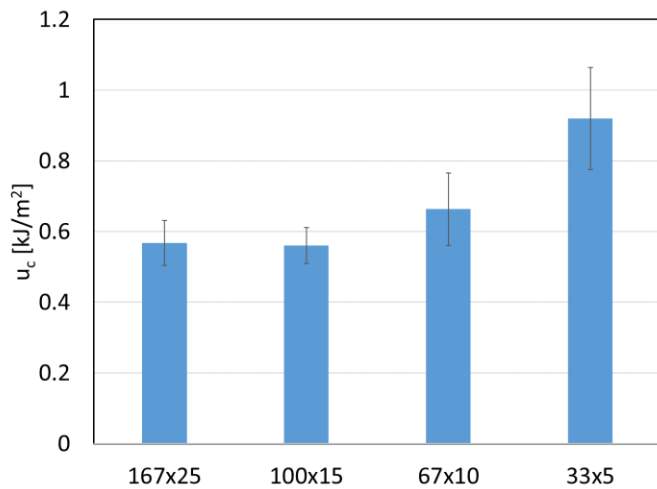


Figure 5: u_c results of SENT specimens with different size (Gillette push and front view).

Also in this case, increasing the specimen dimensions with respect to the standard specimen (100x15) does not alter the fracture toughness, while decreasing the dimensions causes an increase of u_c values, already for the specimen 67x10. Moreover, the results of 67x10 and 33x5 are higher than the results obtained with the same width and standard length (100x10 and 100x5): this could be due to the lower H/W ratio in case of the specimens tested for the size effect.

4 CONCLUSIONS

The present work explores the effects of test conditions and specimen dimensions on fracture toughness of an NBR, measured in quasi-static conditions and at fracture initiation. The aim is to quantitatively define the limit conditions for the obtainment of an intrinsic fracture toughness value.

From tests on the notching method, it could be observed that various notching conditions provide similar mean values but do affect repeatability of u_c results. Instead, two methods of fracture onset monitoring provide very different fracture toughness values, as they seem to identify different physical phenomena related to fracture process: the very begin of damage in case of front view, and, in the case of lateral view, a phase where fracture approaches a stage of constant propagation rate and the sharpening of the blunted notch tip occurs.

From experiments on the influence of specimen dimensions, it was observed that an almost constant value of u_c was obtained for high H/W ratios (above 4.6) and for the larger specimens (W above 10 mm). It seems therefore that for the material investigated, and above these dimensional limits, the measured fracture toughness is an intrinsic material property,

not influenced by specimen dimensions, and that the specimen 70x15 is recommended as the smallest specimen to evaluate this property.

Future works by the authors will focus on validating the limits of geometry dimensions defined in this work to obtain an intrinsic material property by testing other types of rubber, and extrapolating general guidelines useful for the definition of more general requirements.

ACKNOWLEDGEMENT

Semperit Technische Produkte is gratefully acknowledged for providing the rubber material used for the investigation.

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