A Critical Study of an Alternative Method to Measure Cohesive Properties of Adhesive Layers

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Abstract. A perfect experiment is only sensitive to the properties to be analysed. However, evaluation of experimental results is always based on assumptions. Depending on the assumptions, the derived results are more or less correct. In this paper a method based on linear elastic fracture mechanics is compared to a method based on the path independence of the *J*-integral and the assumptions of the existence of a cohesive zone. Contrary to the other methods, the *J*-integral method only rests on the assumption that the material of the specimen has a strain energy density that not explicitly depends on the position in the direction of crack propagation. That is, the conditions for *J* to be path independent. Evaluation of simulated experiments gives the exact value of the fracture energy. The alternative method is based on linear elastic fracture mechanics. Contrary to the conventional methods we use an expression where the crack length is eliminated in favour of the flexibility of the specimen.

Influences of assumptions are studied both experimentally and numerically. Differences in stiffness are achieved by changing the type of adhesive and the layer thickness. Two different adhesives are studied. One is a modern crash resistant epoxy adhesive, *SikaPower-498*. This is a relatively stiff and tough adhesive. The other adhesive is a soft and extremely tough polyurethane based adhesive, *Sikaflex-UHM*. Two layer thicknesses are tested; 1.0 mm for the epoxy and 3.0 mm for the polyurethane based adhesive. The results show that the two methods give similar results for the thinner and stiffer epoxy adhesive but differences are recorded for the soft polyurethane based adhesive. This analysis gives a better understanding of the evaluation methods and their limitations and possibilities to extract cohesive laws.

Introduction

Adhesive layers have two major types of deformation modes, viz. *peel*[1] and *shear*. The stress-deformation relation for each type of deformation mode has to be determined experimentally. Commonly used experiments with stable crack propagation are, the *double cantilever beam* (DCB) specimen for peel, and the *end notch flexure* (ENF) specimen for shear, cf. e.g. [1]. Usually an adhesive layer is stronger in shear than in peel and it is preferably to design joints to take the loading in shear. However, it is common to find that the failure is initiated in peel, cf. e.g. [2]. In the light of these facts the accuracy of the methods used to determine the behaviour of an adhesive layer in peel is important. The geometry of the DCB-specimen is shown in Fig. 1.

Several methods are in use to experimentally determine the peel properties using the DCB-specimen. The methods are based on two principles, *linear elastic fracture mechanics* (LEFM), [3,4] or the path independent *J*-integral [5,6,7]. Most LEFM-methods are based on two assumptions;

[1] Sometimes denoted as *normal stress*

firstly that the flexibility of the specimen is accurately given by elastic beam theory, and secondly, that the process zone in the adhesive layer is confined to a very small area at the crack tip. For the *J*-integral methods, these assumptions are not necessary. However, for the *J*-integral to be path independent, it is necessary to be able to define a strain energy density for the material during loading. It is required that this strain energy density function is not explicitly dependent on the geometrical position in the continuation of the crack path. This can be assumed to be the case if no unloading from an inelastically loaded state occurs.

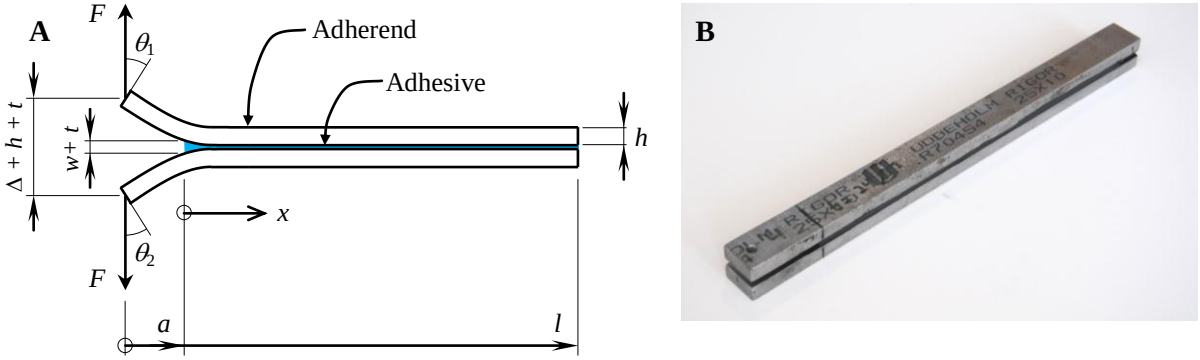

Fig.1. **A**: DCB-specimen with applied loads i.e. the adhereds and the adhesive is deformed, **B**: Photo of DCB-specimen with a 3 mm thick *Sikaflex-UHM* (Polyurethane).

In [8] some commonly used LEFM-methods are analysed with respect to the accuracy. Experiments are simulated using reasonable models of modern tough adhesives. The simulated experimental results are analysed and it is shown that many of these methods give significant errors. The errors depend of the crack length. However, one of these methods shows good accuracy. This method is briefly presented below. It has been noted that this method also gives a good estimation of the energy release rate. It is thereby possible to calculate a stress-deformation relation. Recently, this method has been used to measure cohesive laws for an epoxy adhesive, cf. [9].

Method

Using the *J*-integral approach, the energy release rate for a DCB-specimen is given by,

$$J_{\mathrm{S}} = \frac{F\left(\theta_1 + \theta_2\right)}{b} \tag{1}$$

where F is the applied load, θ_1 and θ_2 are the rotations of the loading points and, b is the width of the specimen, cf. Fig 1. In [8] it is shown that energy release rate can be accurately estimated by,

$$J_{\mathrm{T}} = \frac{F^2}{EIb}\left(\frac{3EI\Delta}{2F}\right)^{\frac{2}{3}} \tag{2}$$

where Δ is the separation of the loading points, E is Young's modulus, and $I \equiv bh^3/12$ is the moment of inertia. This method is based on LEFM. Eq. 2 is derived from beam theory by eliminating the crack length and by use of the compliance Δ/F, cf. [4].

For methods based on the *J*-integral approach it is possible to directly derive the stress-deformation relation for the adhesive layer by differentiating with respect of the peel deformation, w,

$$\sigma = \frac{\partial J}{\partial w} \tag{3}$$

Since Eq. 2 gives a good estimation of the energy release rate, the same method can be used with Eq. 2 although it is based on LEFM. A stress-deformation relation, $\sigma(w)$, usually consists of three parts; a linear elastic increasing stress, a plateau with a constant stress and a softening region. When the stress becomes zero, a macroscopic crack is present and the energy release rate equals the fracture energy, J_c.

Experimental setup

Six experiments with *SikaPower-498*, which is a modern crash resistant epoxy adhesive, and seven experiments with a soft and tough polyurethane based adhesive, *Sikaflex-UHM* are performed. All adherends are made of a tool steel (Uddeholm Rigor) with a yield strength larger than 500 MPa. The epoxy adhesive is cured at 175°C for 25 min. The polyurethane based adhesive is cured by influence of the humidity in room temperature for at least two weeks. A correct layer thickness is achieved by use of PTFE-films with the desired thickness. All experiments are performed with a constant loading rate; for the epoxy adhesive $\dot{\Delta} = 10$ μm/s and for the polyurethane based adhesive $\dot{\Delta} = 33$ μm/s. All the experiments are considered to be quasi static. All the specimens are fixed to the experimental setup by screws. The geometry of the specimens is presented in Table 1.

Table 1. Geometry of the DCB-specimens

Adhesive	a (mm)	l (mm)	h (mm)	b (mm)	t (mm)
SikaPower-498 (epoxy)	80	200	8.0	5.0	1.0
Sikaflex-UHM (polyurethane)	40	340	10.6	25.6	3.0

The tensile test machineries work horizontally. Both the deformation of the adhesive layer and the separation of the loading points are measured with LVDTs. The angles θ_1 and θ_2 is measured in two ways; for the epoxy adhesive with a shaft encoder and for the polyurethane adhesive both with a shaft encoder and with two tilt sensors that are sensitive to the direction of the gravity, cf. Fig. 2. Both types of sensors have high accuracy and resolution. The specimens are oriented vertically in the machinery to reduce the influence of their weight. At the loading points, the specimens are supported by bearings to reduce friction and any applied moments.

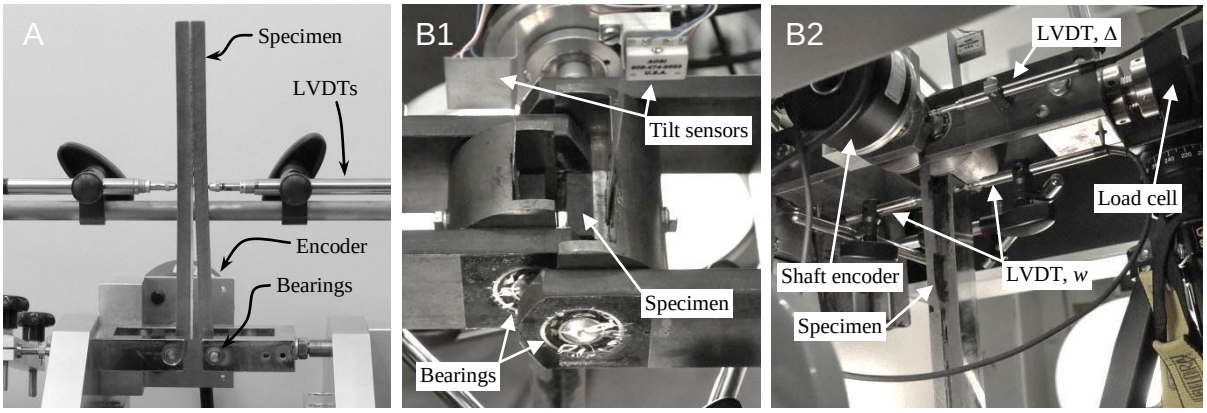

Fig.2. Experimental setup, for the epoxy adhesive. Specimen oriented upwards (A), for the polyurethane adhesive from above (B1) and from below (B2). Specimen oriented downwards.

Experimental result

All experiments are evaluated with both methods i.e. using Eq. 1 and 2. No plastic deformation of the adherends has been observed during the experiments. Typical experimental curves are shown in Fig. 3 where J_S originates from the J-integral method and J_T originates from the LEFM method. The evaluated energy release rate increases until the crack starts to propagate. For all the experiments J_S is lower than J_T. For the polyurethane adhesive the difference is about 10 % and for the epoxy about 5 %, cf. Table 2 and Table 3. Before differentiating using Eq. 3, the scatter in the experimental result of the energy release rate is reduced by use of a first order Butterworth-filter. After this procedure the experimental results are numerically differentiated. Fig. 4 gives the corresponding evaluated stress-deformation relation obtained by Eq. 3. For the polyurethane adhesive it is noted that the largest difference between the methods are obtained during the first part of the plateaus of the curves i.e. for $0.25 < w < 1.5$ mm. During this part, the difference between J_S and J_T are increasing and the difference remains during crack propagation. This has not been observed for the experiments with epoxy adhesive. For the polyurethane adhesive the latter steeply decreasing part of the curves indicates rapid or unstable crack propagation.

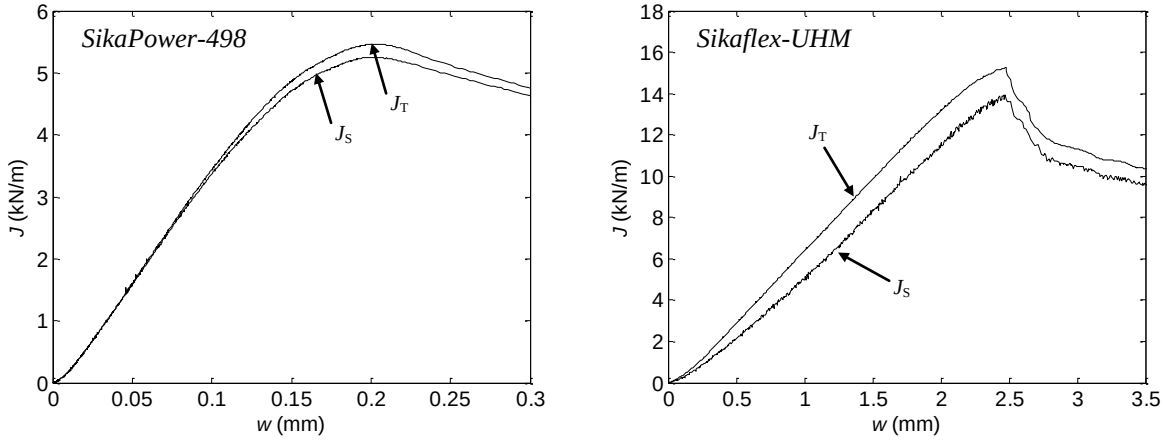

Fig.3. Evaluated energy release rate vs. peel deformation for two experiment, no. 1 and no. 7.

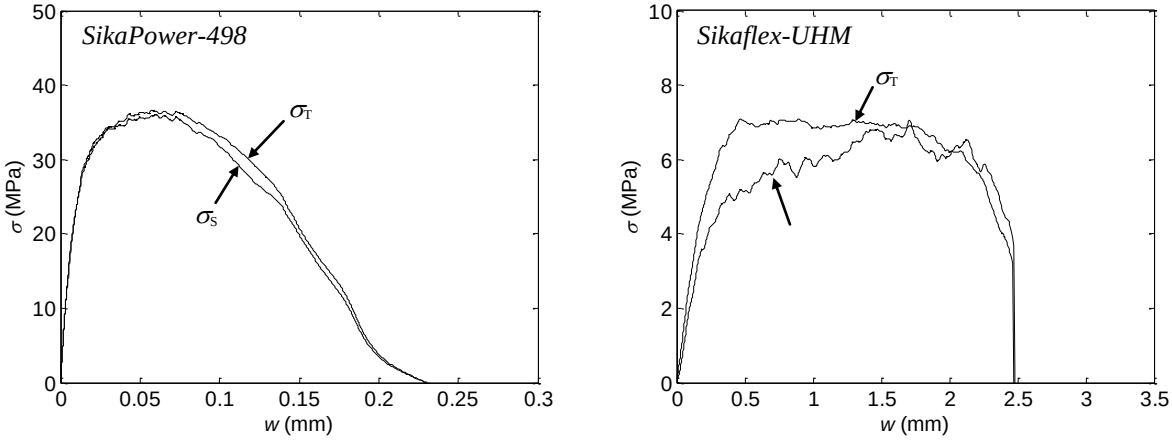

Fig.4. Evaluated stress-deformation relation for two experiment, no. 1 and no. 7.

Table 2. Measured fracture energy and critical deformation for *SikaPower-498* (epoxy)

Specimen no.	**1**	**2**	**3**	**4**	**5**	**6**	**Average**
J_{Sc} (kN/m)	5.08	5.35	5.44	5.41	5.09	4.93	5.22
J_{Tc} (kN/m)	5.28	5.63	5.77	5.56	5.30	5.27	5.46
J_{Tc}/J_{Sc} (-)	1.04	1.05	1.06	1.03	1.04	1.07	1.05
w_c (mm)	0.30	0.30	0.30	0.30	0.30	0.30	0.30

Table 3. Measured fracture energy and critical deformation for *Sikaflex-UHM* (polyurethane)

Specimen no.	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**Average**
J_{Sc} (kN/m)	13.9	18.3	13.5	14.5	15.2	19.5	17.9	16.1
J_{Tc} (kN/m)	15.3	19.2	14.6	15.2	16.5	21.6	20.2	17.5
J_{Tc}/J_{Sc} (-)	1.10	1.05	1.08	1.05	1.09	1.11	1.13	1.09
w_c (mm)	2.48	3.00	2.55	2.45	2.62	2.97	2.70	2.68

Numerical simulation

In order to validate the results, the experiments are simulated using the FE-program *Abaqus*. Only one half of the specimen is simulated. The model consists of beam elements and non-linear spring elements. The force-elongation relation of the springs is adapted to represent the behaviour of the adhesive layer, cf. Fig 5. The length of the beam elements is 0.1 mm. For both types of specimens, the length of the simulated specimen is 300 mm; all the other geometrical parameters are given in Table 1.

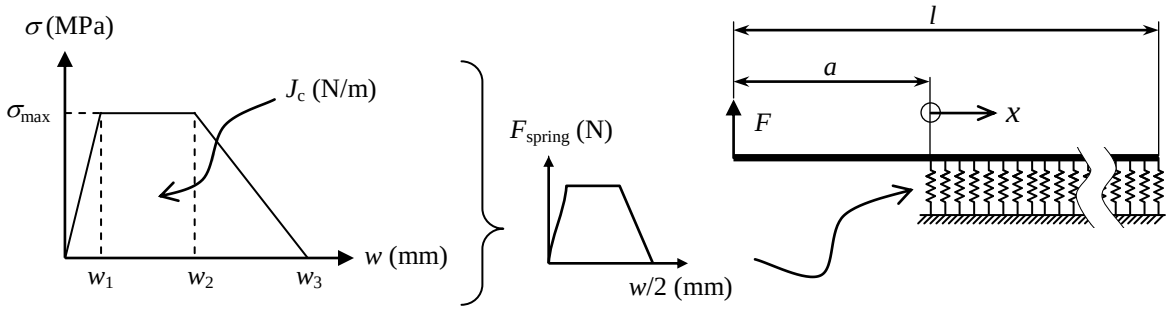

Fig.5 Numerical simulation model where the beam is supported by non-linear springs.

Figure 6 shows the adapted stress-deformation relation used in the simulations (dotted curve) together with the experimentally evaluated results. The results from all experiments are averaged and the scatter is reduced using a first order Butterworth-filter. The experiments are simulated using a stress-deformation relation with three linear parts; the parameters are given in Table 4. The notation is in accordance with Fig. 5.

Table 4. Stress-deformation relation used in the numerical simulations.

Adhesive	σ_{max} (MPa)	w_1 (mm)	w_2 (mm)	w_3 (mm)	J_c (N/m)
SikaPower-498 (epoxy)	34	0.02	0.10	0.23	5270
Sikaflex-UHM (polyurethane)	6.5	0.20	2.50	3.00	16300

In Fig. 7 the simulation (dotted curve) is compared with experimental curves of force, F vs. deformation of the adhesive layer, w, at the start of the adhesive layer. Until the crack starts to propagate, the simulated curve accurately predicts the experimental result. For the polyurethane adhesive the energy release rate decreases, after the crack start to propagate, cf. Fig. 3. This gives a reduced force and the curves in Fig. 7 are diverging. Figure 8 shows the relation between the two

methods of the simulated energy release rates. The difference is significantly larger for the polyurethane adhesive.

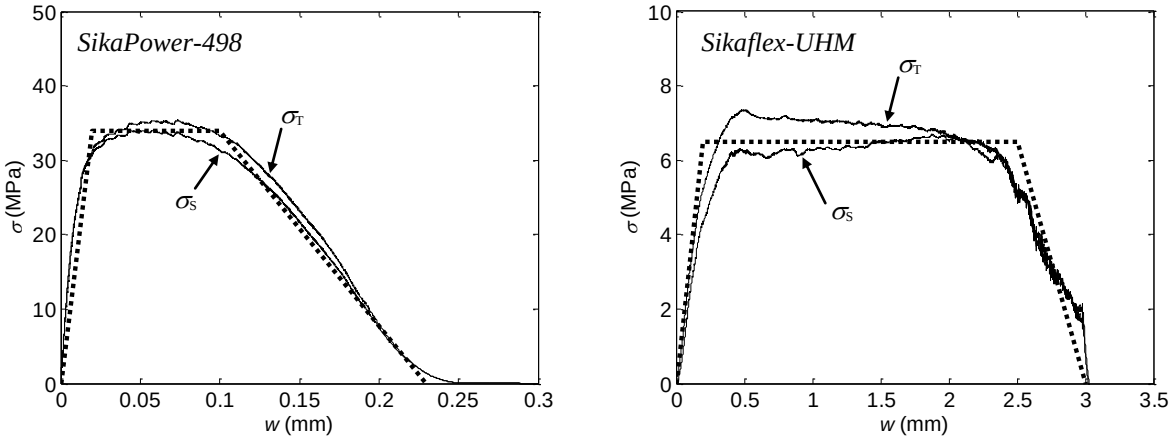

Fig.6. Evaluated experimental results and behaviour used in the simulations (dotted curve).

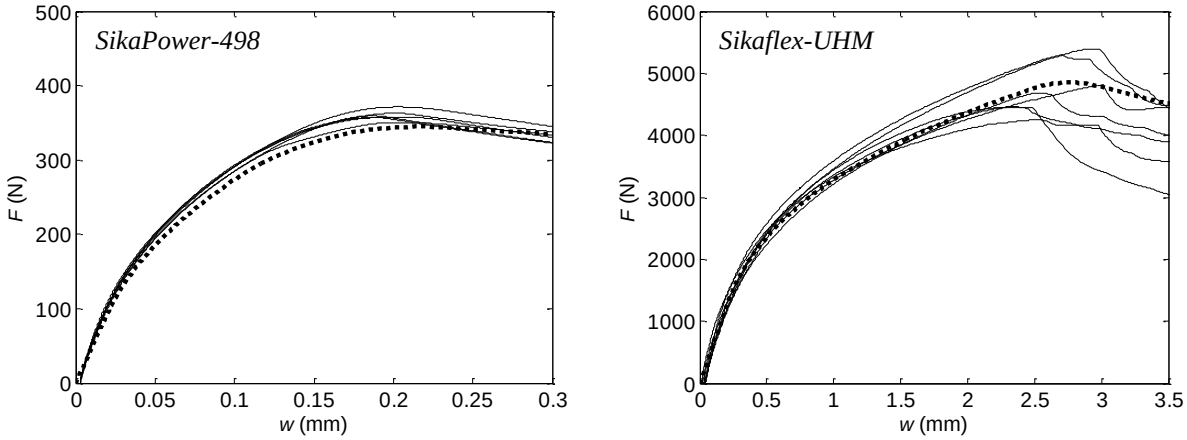

Fig.7. Experimental and simulated (dotted curve) force vs. deformation of the adhesive layer.

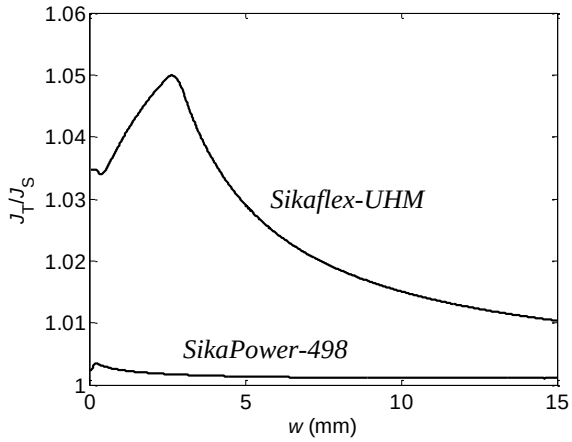

Fig.8. Relation between the energy release rates using the two methods Eq. 1 and 2.

Non-homogenous adhesive layer

During the experiments with the polyurethane adhesive, large necking is observed in the layer prior to crack propagation. The evaluation is based on the assumption of a homogenous adhesive layer. Since the necking varies in the *x*-direction (cf. Fig. 5) this assumption may not be valid. In order to investigate if this influences the evaluation of the energy release rate, two further analyses are performed where the fracture energy for the first 20 mm of the adhesive is changed. The fracture energy is changed by a variation in the maximum stress of the stress-elongation relation, cf. Fig. 9. The energy is linearly increasing (a) or decreasing (b) with the position along the adhesive layer. The analyses are compared with the non-influenced analysis (c) also given in Fig. 8.

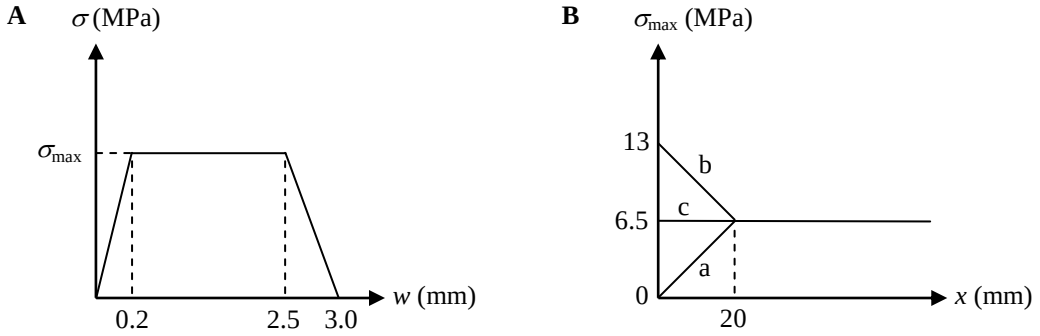

Fig.9. Comparison between simulation and numerical results; A: The stress-deformation relation. B: Variation of σ_{max} according to the position at the specimen.

The results of the analysis are presented in Fig. 10. In all cases $J_T > J_S$ but the difference is always less than 6 %. For all the layers the difference between the methods is decreasing with an increasing crack length.

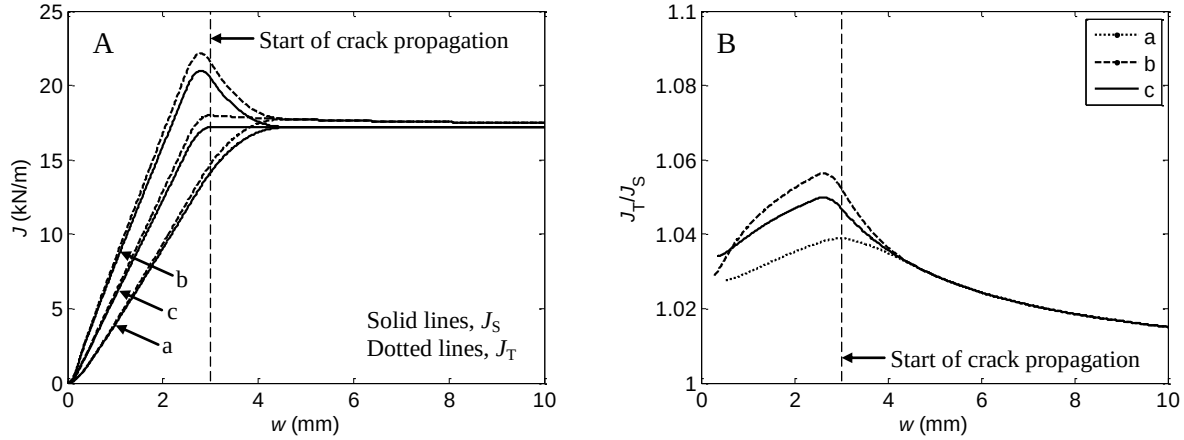

Fig.10. A: Energy release rate vs. deformation of the layer. B: Differences between the methods.

Result and conclusions

Both experimentally and numerically it is shown that the differences between the methods (Eq. 1 and 2) are largest prior to crack propagation. It is also shown that a specimen with a soft adhesive and a short distance between the loading point and the start of the adhesive layer gives a larger error

in the evaluated energy release rate. When measuring stress-deformation relations of an adhesive layer, it is common that the scatter between nominally identical specimens is larger than 6 %. However, since the error is added to the scatter it is important to know the accuracy of the methods. It should also be noted that Eq. 2 is strongly dependent of the geometry and the material properties of the specimens.

It is also shown that increased or decreased fracture energy at the start of the adhesive layer influences the evaluated result. However, it should be noted that the difference observed in the experiments with the polyurethane adhesive is not explained by a difference in the material behaviour by necking at the start of the adhesive layer.

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