Cohesive modelling of the temperature dependence of epoxy based adhesives in Mode I and Mode II loading

by

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Abstract
In this work, the influence of the temperature on the cohesive laws for two epoxy adhesives is studied at temperatures below the glass transition temperature for both Mode I and Mode II loading. Cohesive laws are measured experimentally under quasi-static loading conditions in the temperature range $-30 \leq T \leq 80^\circ\text{C}$. Three parameters of the cohesive laws are studied in detail: the elastic stiffness, the peak stress and the fracture energy. Methods for determining the elastic stiffness in Mode I and Mode II are derived and evaluated. With these methods, the results in this work show that it is possible to measure all three parameters for each pure mode loading case by the use of only the DCB- and the ENF-test specimens. Even though the measures tend to spread in values, this can significantly reduce the cost for performing experiments.

It is shown that most of the cohesive parameters are decreasing with an increasing temperature in both loading modes and for both adhesives. An exception is the Mode I fracture energy for one of the adhesives. This is shown to be independent of the temperature in the studied temperature range. For the same adhesive, the Mode II fracture energy is shown to be continuously decreasing with an increasing temperature.

The experimental results are verified by finite element analyses. The simulations only consider uncoupled cohesive behaviours. By use of the experimental results, simplified bi-linear cohesive laws to be used at any temperature within the studied temperature range are derived for one adhesive in both loading modes. This is desired in order to simulate adhesively bonded structures that suffer a wide range in temperature.

Keywords: Cohesive laws, Epoxy adhesive, Fracture energy, Peak stress, Temperature, Regression analyses, Shear modulus, Young’s modulus.
Acknowledgements
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Tomas Walander
Skövde, March 2013
List of appended papers
This thesis consists of two appended papers.

**Paper A**  

**Paper B**  

Contribution to Co-authored papers
Both appended papers in this thesis are published with my supervisors as co-authors. My contribution to these papers is listed below.

**Paper A**  
Planned and wrote the paper.  
Responsible for the theoretical developments.  
Planned and performed the experiments together with one of the co-authors.  
Evaluated the experiments.  
Responsible for the simulations.  
Responsible for the verifications.

**Paper B**  
Planned and wrote the paper.  
Planned and performed the experiments together with one of the authors.  
Evaluated the experiments.  
Presented the work at the conference
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Introduction

To test a structure experimentally by use of destructive test methods is often an ineffective method that
is associated with high costs. One of the main reason is the fact that it is time-consuming to prepare
and perform the experiments. Another reason for the high costs is that several, nominally identical
experiments need to be performed in order to get reliable results. This is since experimental results
spread. As a substitution, or complement, of full scale experimental tests, it is more cost efficient to
analyse a structure using the finite element method. This becomes obvious when several modifications
and variants need to be tested and/or where the manufacturing cost is high for each tested component.

With the use of adhesives, the performance of a structure can be optimized by enabling joining of
lightweight and high-strength materials. Engineering structures commonly suffer a wide range in
temperature. Since the mechanical behaviour of adhesives is known to be strongly temperature
dependent, cf. e.g. Kinloch (1987), a numerical model of adhesives needs to take temperature into
account as a parameter. No such model has been found in the open literature.

The strength of adhesively bonded, multi-material build-up structures can be adequately predicted
using cohesive elements in a finite element analysis, cf. e.g. Carlberger and Stigh (2010a). The
constitutive relations for these elements are represented with cohesive laws. A cohesive law is a
constitutive relation on a structural length scale between the traction exerted on the interfaces of the
adhesive to the adherends and the separation of the interfaces. The deformation of an adhesive layer
is dominated by two loading modes, cf. e.g. Klarbring (1991) and Schmidt (2008). These are Mode I and
Mode II. Mode I is characterized by peel deformation \( \gamma \) and peel stress \( \sigma \) and Mode II by shear
deformation \( \nu \) and shear stress \( \tau \). An illustration of this is given in Fig. 1.

![Image of cohesive laws](image)

**Fig. 1** An adhesive layer with initial thickness \( t \), cohesive stresses \( (\sigma, \tau) \) with work-conjugated
separations \( (w, \nu) \) and illustrated cohesive laws for pure peel (Mode I) or pure shear (Mode II)
deformation.

Cohesive laws for adhesives can be measured experimentally. For this purpose, two commonly used
test specimens for each loading mode are the double cantilever beam (DCB) and the end notch flexure
(ENF) specimens. By use of Rice’s (1968) and Cherepanov’s (1967), path-independent J-integral,
methods for measuring cohesive laws for these specimens are presented in e.g. Andersson and Stigh
(2004) and Stigh et al. (2009). From these methods, the cohesive laws for an adhesive layer can be
measured in terms of external forces and rotations of the loading points. Experimental results for
adhesive layers using these specimens and methods are presented in e.g. Stigh and Andersson (2000)
and Walander (2009). The methods have the advantage that no material data for the adherends need to
be known when evaluating the experiments. This is as long as the adhesive is assumed as much more
compliant than the adherends. Also, these methods allow for plastic deformation in the adherends as
long as unloading of the specimen is avoided. To allow for plastic deformation can significantly
reduce the size of the specimens in comparison to methods, e.g. Alfredsson (2004) and Tamuzs et al.
(2003), which requires elastic adherends. Small specimens are preferable when performing
temperature experiments since climate chambers often are limited in space.
When evaluating experimentally measured cohesive laws, three parameters are of special interest. These are the elastic stiffness, the peak stress and the fracture energy which are derived from the shape of a cohesive law. The elastic stiffness is defined as the initial slope, the peak stress is defined as the maximum value in stress and the fracture energy is defined as the area beneath the entire curve. For the strength of an adhesively bonded structure, the influence of the elastic stiffness of the adhesive is small. This is since the thickness of an adhesive layer normally is small in comparison to other dimensions. Thus, the deformation of a purely elastic structure is normally dominated by the elastic properties of the bonding material. The strength of an adhesively bonded structure is shown to rather be governed by the parameters peak stress and the fracture energy. Industrially, the elastic stiffness and the peak stress are measured using bulk tensile and thick adherend shear tests. For industrially purposes, several standard methods exist for estimating the fracture energy of adhesive layers by use of linear elastic fracture mechanics (LEFM). For a DCB specimen, some of these methods are evaluated in Biel and Stigh (2008) by use of FE-analysis with a known cohesive law. It is shown that most of the standards, by far, fail to predict the fracture energy. In addition, none of the standardized test methods capture the shape of the cohesive relation. However, methods based on the J-integral both capture the shape of the cohesive law and gives the correct fracture energy.

The influence of temperature on cohesive laws in Mode I is studied by Carlberger et al. (2009). In this study, cohesive laws for the epoxy based DOW Betamate XW 1044-3 (DB1044) adhesive are measured using a DCB specimen. From the study, it is shown that both the parameters peak stress and, to a small extent, the fracture energy are decreasing with an increase in the temperature. In Mode II, no previous study of the temperature dependence for cohesive laws has been reported. However, some studies using LEFM have been performed. Chai (2004) studies the Mode II fracture energy of the American Cyanamid toughened thermosetting (BP-907) epoxy adhesive by use of a Napkin ring specimen. From this study it is shown that the Mode II fracture energy monotonically decreases with an increase in the temperature within the region $0.7 < T/T_g < 1.0$. Banea et al. (2012) study the epoxy based, high temperature, adhesive ChemteX XN1244 using an ENF specimen. The results show a maximum in fracture energy at the temperature $T = 0.88 \ T_g$. In this study, the peak stress at each temperature is not measured. Instead, by use of FE-analysis, the peak stresses are obtained by curve-fitting the numerical results of the force-deformation relation to the experimental results. The procedure is questionable since the influence of the peak stress on the structural behaviour of the ENF-specimen often is small. By performing ENF experiments at several temperatures using the method in Stigh et al. (2009), the influence of temperature for entire cohesive laws and thereby the peak stress and the fracture energy can be measured in Mode II.
Summary of appended papers

Mode I and Mode II experiments are performed on an epoxy based adhesive at different temperatures. The work is performed in a project involving the Swedish automotive industry. This work is the first to report the influence of temperature on cohesive laws in Mode II loading. The main goal is to study the influence of temperature on cohesive laws for an adhesive and to use the results to create a numerical model of the adhesive layer. The adhesive is SikaPower498 (SP498) which is a crash resistant epoxy adhesive that is used today by the automotive industry. The test is performed with a nominal layer thickness of 0.3 mm. Carlberger and Stigh (2010b) show that the thickness of an adhesive layer influences the measured fracture energy in both Mode I and Mode II whilst the peak stress is reported to slightly decrease with an increasing layer thickness in both modes. The used thickness in the present work is decided by the involved industrial project partners. This is with the motivation that it is a common thickness in automotive structures.

The experiments are performed using DCB and the ENF specimens and the experiments are evaluated using the methods in Andersson and Stigh (2004) and Stigh et al. (2009). Experiments are performed at five temperatures for each mode in the span $-40 \leq T \leq 80^\circ C$. This is below the glass transition temperature of the SP498 and the DB1044 adhesive. At least five successful experiments are presented at each of these temperatures and loading modes. Representative cohesive laws for the evaluated temperatures are presented in Paper A, cf. Fig 2. Due to limitations of the climate chambers, the Mode I experiments has a lowest temperature at $-40^\circ C$ and the Mode II experiments $-30^\circ C$.

![Fig. 2 Representative cohesive laws for each temperature group for SP498.](image)

In Paper B, the results in Carlberger et al. (2009) are re-evaluated together with the results of the peak stress and fracture energy for the SP498 adhesive. Regression analysis is used in order to model the influence of the temperature for each cohesive parameter separately. This is done for both adhesives and loading modes. From the evaluation in Paper B, it is shown that the Mode I fracture energy of SP498 can be assumed to be independent of the temperature whilst the other parameters are noticed to monotonically decrease with an increase in the temperature. This is observed for both adhesives and for both loading modes. Another reason to re-evaluate the results in Carlberger et al. (2009) in Paper B is to show that the Mode I fracture energy is not generally independent of the temperature within the evaluated temperature span.

Paper A is a continuation of the work presented in Paper B. In Paper A, two novel methods for determining the elastic stiffness in Mode I using the DCB and in Mode II using the ENF specimen are presented. These methods imply that all three parameters of interest of a cohesive law can be measured
using only two types of specimens. This is without any additional measurements in the experiments. Since the methods are sensitive to small variations of the measured forces and deformations, the experimental results are noticed to spread quite large. However, the methods can still be used for estimating the elastic stiffness. Since the elastic stiffness of an adhesive layer has a small influence on the strength of an adhesively bonded structure, estimations of the elastic stiffness can be sufficient. The presented methods can thus significantly reduce the cost for measuring adhesive properties since no additional tests to determine the elastic stiffnesses needs to be performed.

In Paper A, only the results of the SP498 adhesive is presented for both loading modes. With the results of the peak stress and fracture energy in Paper B, the results of the elastic stiffnesses are also included. Second order regression analyses are performed on all the three cohesive parameters in order to model the influence of the temperature. The elastic stiffnesses are noticed to constantly decrease with an increase in the temperature in Mode I. In Mode II, a minimum of the elastic stiffness is predicted at a temperature of 66°C.

Verification using finite element analysis
Two models for adhesive layers in a FE-code are used in Paper A. These are a shape-mimicking and a bi-linear cohesive law. A shape-mimicking cohesive law has the same shape as a measured cohesive law. It is constructed by defining a damage parameter as a function of the displacement using tabulated data from the representative cohesive laws presented in Fig. 2. The advantage by use of a shape-mimicking cohesive law is that the actual behaviour of an adhesive layer is simulated. That is, no simplifications in shape are made. A disadvantage is that this model only can be used at the temperatures where the cohesive laws have been measured. A bi-linear model, cf. Fig. 3, is a simplified cohesive model. For each mode, it is constructed by the three parameters: the elastic stiffness, the peak stress and the fracture energy. Thus by determining these parameters from experiments, an adhesive layer can be simulated. A great benefit with this model is that, by performing regression analyses of these parameters, any temperature within the evaluated temperature span can be simulated.

![Fig. 3 Bi-linear model with notation for peel and shear](image)

For all evaluated temperatures, simulations of the DCB and the ENF specimens using the bi-linear and the shape mimicking cohesive laws are performed and reported in Paper A. The simulated force vs. load point deformation relations are compared to the experimentally measured relations. A good fit is obtained for nearly all simulations. This gives confidence in the simulated cohesive law. Since the simulated cohesive laws are determined from experiments this also give confidence in the experimental results in Paper A and Paper B. By comparing the outcome of simulations from the analyses using the bi-linear to the shape-mimicking cohesive law, it is shown that the shape of the cohesive law has a minor influence on the structural behaviour for the DCB and the ENF specimens. An exception is at 80°C in Mode II where the bi-linear model is shown to be too much simplified for representing a plateau shaped cohesive law, like the representative cohesive law in Fig. 2.
Intended future work

The results in this work do only consider pure mode loadings. By this, only un-coupled simulations of adhesive layers at different temperatures can be performed. In a FE context, an uncoupled behaviour means that the cohesive laws in one mode are independent the cohesive laws in another. In e.g. a car crash test, a mixed-mode loading of an adhesive layer is common. In order to perform mixed-mode simulations, the mixed mode behaviour needs to be determined experimentally for an adhesive layer. Today there exist only a handful of studies of the mixed mode behaviour of adhesives. By this reason, mixed mode experiments with a prescribed constant mode mix is to be performed by the author.
References


Carlberger T, Stigh U (2010a) Dynamic testing and simulation of hybrid joined bi-material beam. Thin-Wall Str. 48:609-619


Paper A
Temperature dependence of cohesive laws for an epoxy adhesive in Mode I and Mode II loading

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\textbf{Keywords:} Cohesive laws, Epoxy adhesive, Fracture energy, Peak stress, Temperature, Regression analyses, Shear modulus, Young’s modulus.

\section*{Abstract}

The influence of the temperature on the cohesive laws for an epoxy adhesive is studied in the glassy region, i.e. below the glass transition temperature. Cohesive laws are measured in both Mode I and Mode II under quasi-static loading conditions in the temperature range $-30 \leq T \leq 80^\circ\text{C}$. Three parameters of the cohesive laws are studied in detail; the elastic stiffness, the peak stress and the fracture energy. Methods for determining the elastic stiffness in Mode I and Mode II are derived and evaluated. Simplified bi-linear cohesive laws to be used at any temperature within the studied temperature range are derived for each loading mode. All parameters of the cohesive laws are measured experimentally using only two types of specimens. It is shown that most parameters are decreasing with an increasing temperature in both loading modes. The exception is the Mode I fracture energy which is shown to be independent of the temperature. The Mode II fracture energy is continuously decreasing with an increasing temperature. At $80^\circ\text{C}$ the fracture energy is decreased to about $2/3$ of the fracture energy at $-30^\circ\text{C}$. The experimental results are verified by finite element analyses.

\section{1. Introduction}

During the last decades, the automotive industry has attracted focus to both the usage and numerical simulations of adhesive layers in body structures. By using crash resistant epoxy adhesives, combinations of lightweight and tough materials can be considered in order to obtain lighter and more crashworthy automotive body structures. The mechanical behaviour of adhesive layers is studied extensively. In e.g. Kinloch (1987), it is shown that bulk properties of adhesives strongly differ from the results measured using thin films. This shows that the mechanical behaviour of an adhesive depends on the layer thickness. Also the width of an adhesive layer may influence the properties. For a polyurethane adhesive which is much more compliant than epoxies, Biel et al. (2012) show that the maximum measured peel stress is increasing with an increasing width of the specimen. This is explained by the influence of edge boundaries.

By asymptotic analyses Klarbring (1991) and Schmidt (2008) show that a thin adhesive layer of a compliant material can be considered exposed to two main loading modes viz. peel and shear with corresponding peel stress $\sigma$ and shear stress $\tau$, cf. Fig. 1. A cohesive law can represent the stress-deformation relation of an adhesive layer. A cohesive law is a constitutive relation on a structural length scale between the traction exerted on the interfaces of the adhesive to the adherends and the separation of the interfaces. The separation governs the deformation of the adhesive layer. It is denoted $\omega$ and $\nu$ for the peel and shear deformation, respectively. Both loading directions and cohesive laws for each loading mode are sketched in Fig. 1. Cohesive laws for tough epoxy adhesives can be determined experimentally.
However commonly used test specimens like the axially loaded butt joint or the lap shear joint are not applicable for this purpose. Since a cohesive law for an adhesive also includes a softening part, it is necessary that the test specimen suppress instable crack growth. The two most commonly used specimens to measure cohesive laws are the double cantilever beam (DCB) and the end loaded flexure (ENF) specimens. Both geometries give an inhomogeneous stress distribution in the adhesive layer and can be designed to give stable crack growth, cf. e.g. Alfredsson and Stigh (2012).

Fig. 1 Adhesive layer with thickness $t$. Cohesive stresses $(\sigma, \tau)$ with work-conjugated separations $(w, v)$ and typical cohesive laws for pure peel (Mode I) or pure shear (Mode II) deformation

The mechanical properties for polymers vary with temperature. For epoxies, both Young’s modulus and the shear modulus are known to decrease with an increase in the temperature within the glassy region, i.e. at temperatures below the glass transition temperature $T_g$. Both the strain rate and the temperature dependence of cohesive laws for epoxy adhesives have been studied. In Carlberger et al. (2009) it is shown that the epoxy adhesive DOW Betamate XW 1044-3 (DB1044) is both temperature- and strain rate-dependent in Mode I loading for temperatures within the glassy region. The cohesive laws are measured and evaluated at seven equally distributed temperatures with ten repeated experiments at each temperature. It is shown that both the peak stress and, to a small extent, the fracture energy of the cohesive law are decreasing with an increase in the temperature.

No previous study on the temperature dependence of cohesive laws in Mode II loading has been reported. However, the temperature dependence of the Mode II fracture energy is studied by Banea et al. (2012) using linear elastic fracture mechanics (LEFM). They study the fracture behaviour of a 2 mm thick, high-temperature, epoxy adhesive ChemteX XN1244 at the temperatures 20, 100, 150 and 200°C using an ENF specimen. This adhesive has a glass transition temperature of $T_g \approx 428\, \text{K}$. The results show a maximum fracture energy at the temperature $T = 0.88\, T_g$. That is, at 100°C. In the study, the elastic modules of the adhesive at the evaluated temperatures are obtained by bulk tensile- and thick adherend shear-tests. By performing numerical simulations using a bi-linear cohesive law, the peak stresses at the respective temperatures are obtained by curve-fitting the numerical results of the force-deformation relation to the experimental results. It is shown that the peak stresses decrease with an increase in the temperature; but by this approach the peak stress at 200°C is not possible to deduce. Chai (2004) predicts the Mode II fracture energy for an American Cyanamid toughened thermosetting (BP-907) epoxy adhesive according to

$$G_{II,c} \approx t \frac{B_1}{T_g} \left( \frac{t}{t_0} \right)^c \left[ 1 + A_2 \frac{t}{T_g} \log \left( \frac{\gamma_0}{\gamma} \right) \right]$$

where the constants $B_1 = 1011 \, \text{MPa}$, $t_0 = 100 \, \mu\text{m}$, $A_2 = 289 \, \text{MPa}$, $c = 0.21 \, [-]$, $T_g = 371.5 \, \text{K}$ and $\gamma_0 = 10^{34.5} \, \text{s}^{-1}$ are obtained from experiments using a Napkin ring specimen. In this equation, $\gamma$ denotes the shear strain rate and $t$ denotes the thickness of the adhesive layer. The prediction is made from experimental results in the temperature span $0.7 < T/T_g < 1.0$. Figure 2 shows the predicted fracture energy for a 300 $\mu\text{m}$ thick adhesive layer at different shear strain rates. The prediction shows that the Mode II fracture energy decreases continuously with an increasing
temperature at a constant strain rate. Furthermore, Chai (2004) shows that the yield strength decreases with an increasing temperature and increases with an increasing strain rate.

Fig. 2 Prediction of the Mode II fracture energy at constant strain rates [s⁻¹], Chai (2004)

By determining the entire cohesive laws of an adhesive at different temperatures, the strength of an adhesively bonded structure, e.g. an automotive body structure, can be accurately simulated by finite element analyses using the experimental results as input data. An adhesive of current interest to the automobile industries is the crash resistant, thermo-set epoxy adhesive SikaPower498 (SP498). The glass transition temperature is reported by the manufacturer as $T_g \approx 100 \, ^{\circ}C$. The shear strength at room temperature and the influence of the layer thickness are studied by e.g. Walander (2009) and Marzi et al. (2011). The relevant applications for this adhesive in the automotive industry suffer a substantial temperature range. In this paper experiments are performed with this adhesive in Mode I and Mode II within the temperature span $-40^{\circ}C \leq T \leq 80^{\circ}C$. Cohesive laws are measured and statistical methods are used to evaluate the influence of temperature on three parameters: elastic stiffness, peak stress, and fracture energy. For each mode and temperature, these parameters are used to define a simplified bi-linear cohesive law that is used in finite element analyses. Moreover, since the entire cohesive laws are measured in the experiments, a shape mimicking and equivalent cohesive law for each temperature group and loading mode is obtained. The bi-linear and the equivalent cohesive laws are verified by use of finite element analysis. The results are compared to each other and also with the experimental force-deformation relations for verification. The paper ends with some conclusions.

2. Theory and Methods

The cohesive laws are measured with methods utilizing Rice’s (1968) and Cherepanov’s (1967), path-independent $J$-integral,

$$J = \int_C \left( U dy - T \cdot \frac{\partial u}{\partial x} dC \right) \quad (1)$$

where $C$ denotes an arbitrary counter-clockwise integration path, $U$ denotes the strain energy density, $T$ denotes the traction- and $u$ denotes the displacement vector. In this two-dimensional expression, a Cartesian coordinate system is used with the $x$–axis along the adhesive layer. By evaluating Eq. 1 along an integration path encircling a small part of adhesive layer at the crack tip, $J$ is given by

$$J(w, v) = \int_0^w \sigma(\bar{w}, v)d\bar{w} + \int_0^v \tau(w, \bar{v})d\bar{v}. \quad (2)$$
This shows that $J$ is the sum of the strain energy release rate (ERR) under each separate cohesive law shown in Fig. 1. The ERR for a closed, counter-clockwise integration path is zero. By assigning two paths having the same start and end points, Eq. 2 can also be expressed in terms of external loads and rotations. The loads and rotations can be measured experimentally and the ERR of the adhesive layer at the crack tip can thereby be determined. A general, adhesively bonded test specimen is shown in Fig. 3. The specimen can be used for pure mode or mixed-mode loadings. It consists of two adherends that are partially joined by an adhesive layer. The part of the specimen that is not joined by an adhesive layer is considered as a crack with length $a$ and the start of the adhesive layer is denoted the crack tip.

Fig. 3 Deformed mixed mode loaded test specimen having with layer thickness $t$ and out-of-plane width $b$

The specimen is subjected to four applied loads, $F_1$ to $F_4$ and the resulting deformation of the adhesive layer at the crack tip is denoted $w$ for peel- and $v$ for shear-deformation. Clockwise rotations of the adherends, corresponding to the points where the loads are applied, are denoted $\theta_1$ to $\theta_4$. The assigned $J$-integration path for this specimen starts at the crack tip and encircles the boundaries counter clockwise including all the external loads. By having the overhang $c$ large enough, the contribution to the $J$-integral at the right end of the specimen can be neglected. By evaluating the specimen using the $J$-integral with these considerations, $J$ at the crack tip is given by

$$J = \frac{1}{b} (F_1 \sin \theta_1 - F_2 \sin \theta_2 - F_3 \sin \theta_3 + F_4 \sin \theta_4) \tag{3}$$

where $b$ is the width of the specimen. Equilibrium gives $F_2 = -F_3 \frac{d}{L} + F_4$ and $F_4 = F_3 (1 - \frac{d}{L})$. Substitution in Eq. 3 results in

$$J = \frac{F_1}{b} [\sin \theta_1 - \sin \theta_2] + \frac{F_2}{b} \frac{d}{L} \sin \theta_2 - \sin \theta_3 + (1 - \frac{d}{L}) \sin \theta_4. \tag{4}$$

The relation between peel and shear depends on the loading distribution. Two extreme cases can be obtained. The case of pure Mode I loading is obtained when $F_3 = 0$. Symmetry of the specimen then gives that $\theta_1 = -\theta_2$. The result is identical to the ERR for a transversally loaded DCB specimen which is presented in Eq. 5a. The case of pure Mode II loading is achieved when $F_1 = -F_2$ which gives $\theta_1 = \theta_2$. The result is identical to the ERR for an ENF specimen and the result is given in Eq. 5b.

$$J_1 = \frac{2F_1}{b} \sin \theta_1, \quad J_\Pi = \frac{F_2}{b} \frac{d}{L} \sin \theta_2 - \sin \theta_3 + (1 - \frac{d}{L}) \sin \theta_4 \tag{5 a, b}$$

The ERR for a transversally loaded DCB specimen is previously derived in Olsson and Stigh (1989) and with applied moments in Rice (1968) and Suo et al. (1992). Experimental results for structural adhesives using these methods are presented in e.g. Stigh and Andersson (2000), Sørensen (2002), Andersson and Stigh (2004) and Andersson and Biel (2006). Similarly, methods for the ENF specimen are derived and experimental results are presented in Alfredsson et al. (2003), Alfredsson (2004), Leffler et al. (2007) and
Stigh et al. (2009) with applied forces and in e.g. Martin et al. (1999) with applied moments by using a four point bend ENF specimen. For transversal loads, Alfredsson (2004) derive the ERR for a symmetrical, transversally, loaded (i.e. \( d = \frac{L}{2} \) in Fig. 3) ENF specimen as

\[
J_{II} = \frac{9}{16} \frac{F_3^2 a^2}{E_a b^2 h^3} + \frac{3 F_3 v}{8 b h} - \frac{9}{128} \frac{F_3^2}{K_s b^2 h^2}
\]  

where \( K_s \) denotes the elastic shear stiffness of the adhesive layer and \( E_a \) denotes Young’s modulus of the adherends. This method has been shown to capture cohesive laws; however with some conditions. For Eq. 6 to be valid the adherends must remain linear-elastic during the experiment and the process zone of the adhesive must be accommodated within the space between the crack tip and the loading point. Moreover, according to LEFM, an ENF specimen tends to be unstable for crack lengths shorter than \( a < 0.35 L \), cf. Carlsson et al. (1986) and Alfredsson and Stigh (2012). This requires the length of the specimen to be larger than about 6.7 times the length of the adhesive’s process zone. The length of a process zone is in Stigh et al. (2010) estimated with

\[
L_{II,p} = \frac{E_a h}{12 J_{II,c}}
\]

where \( v_c \) denotes the critical shear deformation and \( J_{II,c} \) denotes the Mode II fracture energy of the adhesive. For a \( t = 0.36 \) mm thick SP498 adhesive, experimental results at room temperature by Walander (2009) gives \( v_c = 482 \) µm and \( J_{II,c} = 12.9 \) kJ m\(^{-1}\). If this adhesive should be evaluated by use of Eq. 6, it is required that \( h = b = 30 \) mm in order to ensure that the adherends remain elastic with the yield strength 500 MPa for the adherends. This results in a specimen length of \( L \geq 0.63 \) m. The required maximum load for an experiment with this specimen is predicted by

\[
F_{3,c} = \frac{4b}{3a} \sqrt{E_a h^3 J_{II,c}} \approx 111 \text{ kN}, \text{ cf. Alfredsson (2004).}
\]

Thus, an inconveniently large specimen and thereby last testing machine is required for the present adhesive.

A smaller test specimen is allowed for if Eq. 5b is used instead of Eq. 6 in the evaluation. The \( J \) integral given by Eq. 1 is valid for any non-linear elastic material as long as the strain energy density \( U \) can be defined and if \( U \) is not explicitly dependent on the coordinate \( x \), cf. e.g. Nilsson (2001). Before unloading, there is no difference in mechanical response for nonlinear elastic and an inelastically deformed material. This implies that Eqs. 3 to 5 are valid for specimens that deform plastically under the condition that unloading from a plastic state is avoided. To allow for plasticity gives some advantages. The greatest advantage is that the geometry of the specimen and thereby the required load can be significantly reduced. Moreover, the risk for unstable crack propagation is decreased due to the yielding of the specimen. Another advantage is that the crack length does not need to be known with precision since it does not enter Eq. 5b. Since the crack length in Eq. 6 is raised to a power of two, an error in measurement of this parameter gives a large influence on the measured ERR. The disadvantages with having a smaller, plastically deformed, specimen is that it becomes more sensitive to local disturbances in the adhesive layer at the crack tip and that the adherends cannot be re-used in future experiments. It should also be noted that during crack propagation, unloading from a plastic state cannot be avoided. However, when measuring cohesive laws, the ERR of the adhesive layer at the crack tip is measured to the point before crack propagation and thus the unloading has a minor influence.

When performing ENF experiments, the friction between the adherends influences the measured ERR. The effect of friction has been studied previously by use of finite element analyses. In Mall and Kochhar (1986) the difference in measured Mode II fracture energy for a specimen with a crack length of \( a = 0.35 L \) is estimated to be between 0.6 to 2 % for a sliding friction coefficient of \( 0.1 \leq \mu_s \leq 0.3 \) as compared to a specimen without friction. In Davidson and Sun (2005), the difference is shown to be less than 3.6 % for \( \mu_s = 0.3 \). By using LEFM and Euler-Bernoulli beam theory, Carlson and Gillespie (1986, 1989) derive a non-dimensional increase in measured ERR for an ENF specimen as

\[
p(\mu_s) = \frac{3}{4} \mu_s \frac{h}{a}
\]

For the dimensions
used in this work, cf. Table 1, $p(\mu_s) = 3.4\%$ for $\mu_s = 0.3$. By inserting a polytetrafluorethylene (PTFE) film between the adherends, the sliding friction coefficient is reduced to $\mu_s \equiv 0.15$ which gives $p(\mu_s) = 1.7\%$. It is the applied force, $F_3$ in Fig. 3, that gives rise to the friction forces between the adherends. The previous studies only consider ENF specimens with linear elastic adherends. A specimen having adherends that are allowed to yield requires a smaller applied force to create fracture than a specimen having elastic adherends. By this it is assumed that the error in measured Mode II ERR due to friction is smaller for specimens that yield. Therefore the influence of friction is neglected in all evaluations in this work.

2.1 Elastic stiffness of adhesive layer

In Stigh (1988) the deformations in a DCB specimen having a bi-linear constitutive relation is analytically derived by use of beam theory. In that work symmetry is used where $E_a l_e = \frac{1}{2} E_a \frac{bh^3}{12}$ denotes the equivalent bending stiffness of the beams. By using a coordinate $x$ starting at the crack tip the total, relative, displacement between the beams $w(x)$ is described by the equilibrium equation $E_a I_e w(x)'' + q(w(x)) = 0$ where $q$ denote the load per unit length exerted by the local deformation of the adhesive. For a linear elastic adhesive, $q = K_n w(x)$ where $K_n$ denotes the elastic peel stiffness of the adhesive. The solution to the equilibrium equation is $w(x) = e^{-\lambda x} A_1 \sin \lambda x + A_2 \cos \lambda x$ with $\lambda^4 = \frac{4 K_n}{E_a h^3}$. Until damage initiates at the crack tip the specimen is linear elastic. At damage initiation, the deformation at the crack tip ($x = 0$) and the corresponding load level is denoted $w_i(x = 0)$ and $F_1$ respectively. The integration constants $A_1$ and $A_2$ are then determined by the transversal shear force and the bending moment at the crack tip as $F_1 = E_a I_e w_{i1}$ and $F_1 a = E_a I_e w_{i1}$. From the theory in Stigh (1988) the deformation at the crack tip is expressed as

$$w(x = 0) \equiv A_2 = \frac{E_a I_e}{E_a b h^3 \lambda^2} (\lambda a + 1) \quad \text{for } 0 \leq w < w_i$$

(7)

with $\lambda^4 = \frac{6 K_n}{E_a h^3}$ and the geometry corresponding to Fig. 3. Eq. 7 holds for any deformation up to damage initiation. For $\lambda a \gg 1$ the elastic peel stiffness is obtained as

$$K_n = \frac{24 a^2}{E_a b h^3} \left( \frac{F_1}{w} \right)^2 \quad \text{for } 0 \leq w < w_i$$

(8)

or, generally,

$$K_n = \frac{E_a h^3}{6} \left\{ \frac{36}{E_a b h^6} \left( \frac{F_1}{w} \right)^2 - \frac{64 a^3}{E_a b h^9} \left( \frac{F_1}{w} \right)^3 + \frac{6}{E_a b h^3} \left( \frac{F_1}{w} \right)^4 \right\}$$

$$\ldots + \frac{4 a}{E_a b h^3} \left( \frac{F_1}{w} \right)^4 \quad \text{for } 0 \leq w < w_i.$$
\[
\tau = K_s \nu \approx \bar{\tau}(\kappa a + 1)
\]

where

\[
\begin{aligned}
\bar{\tau} &= \frac{3 F_3(1-\eta)}{2 b(2h)} \\
\kappa &= \sqrt{\frac{6K_s}{E_s h}}.
\end{aligned}
\]

(10)

In Eq. 10, \(\kappa\) denotes a wave-number to the solution and \(\bar{\tau}\) denote the maximum transversal shear stress predicted by Jouravski’s beam theory for transverse shear stresses in solid beam sections. This maximum is given as \(\bar{\tau} = \frac{3V}{2A}\). Here \(V = F_3(1-\eta)\) denotes the shear force in the beam at the crack tip and \(A = 2bh\) denotes the cross sectional area of the beam at this location. Solving for \(K_s\) in Eq. 9 yields

\[
K_s \approx \frac{9\eta^2}{2} \frac{a^2}{E_a b^2 h^3} \left( \frac{\varepsilon}{\nu} \right)^2 \equiv \bar{K}_s
\]

for \(\kappa a \gg 1\) (11)

or generally,

\[
K_s = \frac{1}{4} \bar{K}_s \left[ 1 + \sqrt{1 + \zeta} \right]^2
\]

where \(\zeta = \frac{2}{5\eta} \frac{E_a h^3}{a^2} \left( \frac{\bar{\tau}}{\nu} \right)\). (12)

Experimentally, the relations between the acting force and the measured deformation, i.e. \(F_1\) vs. \(w\) and \(F_3\) vs. \(v\), contain scatter at the lower loads. Using linear regression, the slopes of the relations \(F_1/w\) and \(F_3/v\) are evaluated. The slopes are used in Eqs. 9 and 12 to derive \(K_o\) and \(K_s\), respectively.

### 2.2 Statistical methods

In this work, regression analyses are used to evaluate the temperature dependence of the elastic stiffness, the peak stress, and the fracture energy for each loading mode. These are collectively denoted response variables, \(y\). The influence of one explanatory variable \(x_1\), here the temperature, is modelled by a linear and a second order model, i.e.

\[
y = x_1 \beta_1 + \beta_0 + \varepsilon, \quad y = x_1^2 \beta_2 + x_1 \beta_1 + \beta_0 + \varepsilon.
\]

(13 a, b)

where \(\varepsilon\) is denoted the disturbance term. The models in Eq. 13a and Eq. 13b does not account for other possible explanatory variables such as e.g. the strain rate or the layer thickness of the adhesive. By estimating the parameters \(\beta_2\), \(\beta_1\) and \(\beta_0\) by the least square method, the influence of the explanatory variable is predicted. Estimated parameters are given by \(\hat{\beta}_2\), \(\hat{\beta}_1\) and \(\hat{\beta}_0\). When performing this estimation the explanatory variable is assumed to be measured without errors. That is, the temperature is assumed to be measured exactly. The disturbance term is estimated to be zero. If the influence of both \(\beta_2\) and \(\beta_1\) is small, the response variable may be considered independent of the explanatory variable. A measure if an estimated regression curve gives a good fit with the measured data is the coefficient of determination \(R^2\). Two extreme values of \(R^2\) are possible. If \(R^2 = 1\), the fitted curve passes through all data points, i.e. the model fits the data exactly. On the other hand, if there is no dependence of the response variable on the explanatory variable, \(R^2 = 0\), cf. e.g. Johnson and Wichern (1992). With \(R^2\) close to zero we can assume an independence of the explanatory variable.

Another way of analysing independence is to use the Kruskal-Wallis test, cf. Kruskal and Wallis (1952). This is a one-way analysis of variance of ranks that enables testing several populations against each other simultaneously. A population is all samples, i.e. measurements, at one specific temperature. This gives \(g\) populations corresponding to \(g\) number of evaluated temperatures. All observations are ranked independent of the temperature. That is, the smallest value is given the rank 1; the second smallest is given the rank 2 and so on until the largest value is given the rank \(n\) equal to the total number of observations. The test is a non-parametric rank test that unlike regression analysis, does not assume a normal distribution. Instead, if
the number of samples of each population $n_i$ is large enough, a certain test variable $K$ can be assumed to be $\chi^2$-distributed. If $K < \chi^2_{g-1}$ the medians of each population are assumed equal. Thus, indicating that there is no dependence of the explanatory variable. In each population, the average rank of the observations is calculated $\bar{r}_i$ where $i = 1, 2, \ldots, g$. The test variable $K$ is calculated according to

$$K = \frac{12}{n(n+1)} \sum_{i=1}^{g} n_i \bar{r}_i^2 - 3(n + 1)$$

(14)

with $n_i$ equal to the number of observations in sample $i$. The probability that the medians are equal is estimated using a table of the $\chi^2$-distribution. More formally, the null hypothesis that there is no difference in the response variable between the samples is rejected if $K \geq \chi^2_{g-1}$. This risk of rejecting the null hypothesis even if it is true is denoted the level of significance. With a 5% risk, a value of $K_{g=5} < 9.49$ is needed to reject the null hypothesis for five evaluated temperatures.

3. Experiments

Test specimens are manufactured with the dimensions according to Table 1. The adherends are made of Uddeholm Rigor which is a tooling steel having the yield stress $\sigma_y > 500$ MPa, Young’s modulus $E_a = 190$ GPa and Poisson’s ratio 0.33. This steel is chosen due to its high yield strength and also since it allows for high engineering strain until the stress is degrading, cf. Walander (2009). This is preferable when using the $J$-integral where unloading is desired to be kept to a minimum. The test specimens are manufactured from bonded Rigor steel plates which are cut to specimens using a band saw. Each plate gives eight to ten specimens. The start of the adhesive layer and also the nominal thickness of $t = 0.3$ mm are achieved by PTFE films. Before cutting, the plates are cured individually at 175°C for 25 minutes. After cutting, both sides of the specimens are face milled to the correct width with cut depths each less than 0.2 mm, with a low feed rate and without coolant. Thus, the milling operation is assumed not to damage the adhesive layer.

### Table 1 Specimen dimensions corresponding to Fig. 3.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>$a$ (mm)</th>
<th>$b$ (mm)</th>
<th>$c$ (mm)</th>
<th>$d$ (mm)</th>
<th>$h$ (mm)</th>
<th>$L$ (mm)</th>
<th>$t$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DCB</td>
<td>80</td>
<td>5.0</td>
<td>-</td>
<td>-</td>
<td>6.6</td>
<td>160</td>
<td>0.3</td>
</tr>
<tr>
<td>ENF</td>
<td>70</td>
<td>10.6</td>
<td>50</td>
<td>60</td>
<td>10.6</td>
<td>200</td>
<td>0.3</td>
</tr>
</tbody>
</table>

In addition to the dimensions presented in Table 1, an overhang of 20 mm is used on the left end of the ENF specimens in order to secure that the specimens remain on the support during deformation. This overhang is shown in Fig. 4. The experiments are performed in the temperature span $-40 \leq T \leq 80$°C. The aim is to test the adhesive in both Mode I and Mode II at five equally distributed temperatures within this span. However, the climate chamber used for the ENF test rig has a lower limit of -30°C. This temperature is thus the lowest evaluated temperature for the Mode II experiments. The influence of temperature on the mechanical properties of the steel is assumed to be negligible in the evaluated temperature span.

All specimens are given ID numbers denoting the plate number, the position of the plate and at which temperature the specimens is evaluated. Specimens located at the outermost of a plate might have other mechanical properties than specimens located at the centre of the same specimen plate, e.g. due to an
uneven temperature distribution during curing. Also entire plates might have defects that influence the mechanical strength. To minimize the influence, specimens from all specimen plates are randomly distributed for testing at different temperatures.

![Specimen](image1)
![LVDTs](image2)

Fig. 4 Experimental set-ups with deformed specimens. Left: DCB test rig. Right: ENF test rig

The experimental set-ups are shown in Fig 4. The climate chambers are not shown in these images. For the DCB testing machine, two horizontally working ball screws are separating the load points symmetrically with a prescribed loading rate of \( \Delta \sigma = 10 \, \mu \text{m/s} \). This is slow enough to consider the Mode I experiments quasi-static. Since the specimen is assumed to deform symmetrically, only the rotation \( \theta_1 \) is measured using a shaft encoder with the resolution \( \pi \cdot 10^{-5} \text{ rad/pulse} \). This assumption has been tested in earlier experiments and is found to give sufficient accuracy. During the experiments, the peel deformation \( \omega \) and the load point displacement \( \Delta \) are measured using linear variable differential transformers (LVDTs), each with a resolution of 1 \( \mu \text{m} \). The peel deformation is measured on the outside of the specimens, cf. Fig. 4. This is possible since the adhesive is much more compliant than the adherends. Finite element analyses of earlier experiments show that this procedure gives sufficient accuracy, as long as the LVDTs are correctly positioned.

For the ENF set-up, a servo hydraulic test machine is used. The test rig has a frame and a load cell designed for loads up to 100 kN. The load cell has an inaccuracy of less than 0.5 %. By knowing the horizontal position between two vertical mounted LVDTs, cf. Fig. 4, measurements of each rotation \( \theta_i \) where \( i = 1, 3 \) and 4 are obtained, cf. Fig 3. The distances are 70 mm for the middle and 50 mm for the other measurements. Also one LVDT for measuring the shear deformation \( v \) is used. The load point displacement \( \Delta \) is measured internally in the test machine and is given a prescribed rate, \( \Delta \sigma = 0.5 \, \text{mm/min} \). This is also slow enough to consider the experiments as quasi-static. All measurement equipment used in the ENF setup is calibrated before performing any experiment. In conjunction to the calibration, the compliance of the ENF test rig is determined. The compliance is not shown to have any effect on the measurements of the load point displacement \( \Delta \) and can therefore be neglected. For the DCB-experiments, the load point displacement is measured externally and thus the compliance of the test machine does not need to be accounted for. Although the loading rate, \( \Delta \sigma \), is constant both for the Mode I and Mode II experiments, the strain rate in the adhesive at the crack tip is not constant during the experiment. This is a result of the non-linear behaviour of the adhesive. However these strain rates are believed to be low enough to also be considered as quasi-static.

4. Experimental results

For convenience, \( F_1 \) for the DCB and \( F_3 \) for the ENF specimen are from now on replaced with \( F \) to denote the force at the loading point for each specimen. Similar, the load point displacements for the specimens
are denoted $\Delta$. Experimental results of the force vs. load point displacement are given for each temperature and loading mode in Fig. 5. It is noted that all experiments result in stable crack propagations. For Mode I loading the experimental results indicate that the maximum force is constant with respect to the temperature while the maximum force is decreasing with an increase in the temperature in Mode II. For both modes, the deformation at the point of maximum force is increasing with an increase in the temperature.

The cohesive laws are obtained by differentiating the measured ERR, given by Eq. 5a and Eq. 5b, with respect to the pure mode deformations, $w$ and $v$ respectively. The experimental results contain scatter and therefore the ERR vs. the pure mode deformation curves are represented by adapted Prony series, cf. e.g. Fernberg and Berglund (2001). The adaptions are differentiated and the results are shown by thin red lines in Fig. 6.

For both modes the peak stress is decreasing for an increasing temperature while the point of maximum deformation is increasing with an increase in the temperature. For Mode I the deformation at the peak stress is virtually constant at about 15 $\mu$m. For Mode II the deformation at this point is more dependent on the temperature. For the lower temperatures, the deformation at the peak stress is about 100 $\mu$m and for the temperature $T = 80^\circ$C the peak stress is achieved at a deformation of about 400 $\mu$m. The curves in Fig. 6 are cut when the stress has decreased to 10 % of the peak stress value.

### 5. Evaluation

All specimens are carefully investigated after the experiments. Both the DCB and the ENF specimens are noted to have remaining plastic deformations after the experiments. Every specimen is bended apart after the experiments and the fracture surface are examined. During manufacturing of the specimens, air bubbles may be captured in the adhesive layer. Specimens with air bubbles in the region close to the crack tip are excluded from the evaluation since it affects the measured fracture energy. Only experiments without visible defects are presented. The number of evaluated experiments is shown in Table 2. By excluding specimens with defects, an ideally adhesive layer is evaluated.

**Table 2** Number of evaluated experiments at each temperature group, $n_i$.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>$T = -40^\circ$C</th>
<th>$T = -30^\circ$C</th>
<th>$T = -10^\circ$C</th>
<th>$T = 20^\circ$C</th>
<th>$T = 50^\circ$C</th>
<th>$T = 80^\circ$C</th>
</tr>
</thead>
<tbody>
<tr>
<td>DCB</td>
<td>10</td>
<td>-</td>
<td>9</td>
<td>6</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>ENF</td>
<td>-</td>
<td>8</td>
<td>5</td>
<td>7</td>
<td>7</td>
<td>6 a)</td>
</tr>
</tbody>
</table>

a) Adhesive fractures

The type of fracture is determined from the fracture surfaces. All evaluated specimens in Table 2 have cohesive fracture surfaces with the exception of the Mode II experiments performed at $T = 80^\circ$C. All of these have adhesive fractures, i.e. a loss in adhesion to the adherends. Since the specimens are ID marked it is concluded that this is not due to defects in the manufacturing procedure. It should however be noted that the strength parameters for these experiments measure the properties of the interface and not the properties of the adhesive layer. All Mode I experiments performed at this temperature show cohesive fracture surfaces. It is thus concluded that the SP498 adhesive tends to be more sensitive to adhesive failure in Mode II at the high temperature.
Fig. 5 Force vs. load point displacements. *Left*: Mode I. *Right*: Mode II. *Thin red lines*: Experimental results. *Thick solid black lines*: FE-results using a full average cohesive law. *Thick dotted and thick dashed black lines*: FE-results using a bi-linear cohesive law obtained from each temperature groups mean value and second order regression analysis, respectively.
Fig. 6 Cohesive laws. Left: Mode I. Right: Mode II. Thin red lines: Experimental results. Solid thick black lines: Full, average, cohesive law. Thick dotted and thick dashed thick black lines: Bi-linear cohesive law obtained from each temperature groups mean value and bi-linear cohesive law obtained from second order regression analysis respectively.

For each experiment the elastic stiffnesses are calculated using Eq. 9 and Eq. 12 for each mode respectively. The results are presented in Fig. 7. The right figure is cropped and one measurement of $T = -30^\circ\text{C}$ is not shown. This result is high in comparison to the other results performed at
the same temperature. Since no obvious error in measurements is detected for this experiment, the result is still included in the further evaluation. The elastic stiffness depends on the thickness of the adhesive layer. For engineering purposes, it is more favourable to measure elastic constants which do not depend on the thickness. Such constants are the effective Young’s modulus, $\overline{E}$, and the shear modulus, $G$. These are related to the elastic stiffnesses according to $K_n = \frac{\overline{E}}{t}$ and $K_s = \frac{G}{t}$. The results of the elastic modules are also presented in Fig. 7.

![Fig. 7](image1.png)

**Fig. 7** Experimental results of elastic stiffnesses and the elastic modules vs. temperature. *Left:* Mode I. *Right:* Mode II. The solid lines combine the mean value of each temperature group. The dashed and the dashed-dotted lines show the results of a first and a second order regression analysis, respectively.

The two cohesive parameters peak stress and fracture energy are obtained from the cohesive laws shown in Fig. 6. The peak stress is determined by the maximum value of each curve. It is shown in Fig. 8. The fracture energy is determined as the point on the $j$-curve corresponding to the point on Fig. 6 where the stress has decreased to 10% of its peak value. This is done individually for each experiment. The results are shown in Fig. 9.

![Fig. 8](image2.png)

**Fig. 8** Experimental results of peak stress vs. temperature. *Left:* Mode I. *Right:* Mode II. The solid lines combine the mean value of each temperature group. The dashed and the dashed-dotted lines show the results of a first and a second order regression analysis, respectively.
Fig. 9 Experimental results of fracture energy vs. temperature. Left: Mode I. Right: Mode II. The solid lines combine the mean value of each temperature group. The dashed and the dashed-dotted lines show the results of a first and a second order regression analysis, respectively.

For each temperature group, the average values of the results in Figs. 8 to 10 are calculated. These are shown by solid lines in the figures and are also presented in Table 3. By performing regression analyses, the estimated trends with respect to the temperature are calculated according to Eq. 13a and Eq. 13b using the least square method. The estimated coefficients of regression are presented in Table 4 where the temperature in centigrade is used. In order to determine if there is a linear trend of the variables, a linear regression line is visualized by dashed lines in Figs. 8 to 10. The result of the second order regression analyses are presented in Table 5 and also by dashed-dotted lines in Figs. 8 to 10. For values of $\tau$ and $J$ in Table 3 to 5, the values of $K_n$ and $K_s$, respectively, should be multiplied with the thickness $t = 0.3$ mm.

Table 3 Experimental results for each temperature group, $n_i$.

<table>
<thead>
<tr>
<th>Average value of</th>
<th>Temperature, $T$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-40°C</td>
</tr>
<tr>
<td>$K_n$ [TN m$^{-3}$]</td>
<td>13.5</td>
</tr>
<tr>
<td>$K_s$ [TN m$^{-3}$]</td>
<td>-</td>
</tr>
<tr>
<td>$\sigma_{\text{max}}$ [MPa]</td>
<td>58.2</td>
</tr>
<tr>
<td>$\tau_{\text{max}}$ [MPa]</td>
<td>-</td>
</tr>
<tr>
<td>$J_{lc}$ [kN m$^{-1}$]</td>
<td>2.84</td>
</tr>
<tr>
<td>$J_{\text{Ilc}}$ [kN m$^{-1}$]</td>
<td>-</td>
</tr>
</tbody>
</table>

$^a$ Walander (2009) and Marzi et al. (2011) obtained $\tau_{\text{max}} \approx 38$ MPa.

$^b$ Walander (2009) and Marzi et al. (2011) obtained $J_{\text{Ilc}} \approx 12.9$ kN m$^{-1}$. 
Table 4 Estimated coefficients of regression and test value, $K$.

| Mode | $\hat{\beta}_2$ [GN m$^{-3}$ °C$^{-2}$] | $\hat{\beta}_1$ [GN m$^{-3}$ °C$^{-1}$] | $\hat{\beta}_0$ [TN m$^3$] | $R^2$ [-] | $\hat{\beta}_2$ [kPa °C$^{-2}$] | $\hat{\beta}_1$ [kPa °C$^{-1}$] | $\hat{\beta}_0$ [MPa] | $R^2$ [-] | $K$ [TN m$^{-3}$] | $\hat{\beta}_2$ [N m$^{-1}$ °C$^{-2}$] | $\hat{\beta}_1$ [N m$^{-1}$ °C$^{-1}$] | $\hat{\beta}_0$ [kN m$^{-1}$] | $R^2$ [-] |
|------|-------------------------------------|-------------------------------------|----------------------|--------|--------------------------------|--------------------------------|----------------------|--------|----------------|--------------------------------|--------------------------------|--------------------------------|----------------------|--------|
| I    | 0.09                                | -28.2                               | 2.93                 | 0.65   | -1.08                          | -270                          | 50.6                 | 0.88   | -1.91          | 1.28                          | 2.93                          | 0.05                          | 6.04                 |        |
| II   | 1.47                                | -194                                | 7.28                 | 0.65   | 1.53                           | -439                          | 38.8                 | 0.92   | -0.28          | -21.4                         | 8.66                          | 0.57                          | 17.2                 |        |

$^a$) Second order regression analysis
$^b$) Linear regression analysis

Table 5 Results from the second order regression analyses.

<table>
<thead>
<tr>
<th>Estimated value of</th>
<th>Temperature, $T$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-40°C</td>
</tr>
<tr>
<td>$K_n$ [TN m$^3$]</td>
<td>13.9</td>
</tr>
<tr>
<td>$K_s$ [TN m$^3$]</td>
<td>17.4</td>
</tr>
<tr>
<td>$\sigma_{max}$ [MPa]</td>
<td>58.8</td>
</tr>
<tr>
<td>$\tau_{max}$ [MPa]</td>
<td>58.7</td>
</tr>
<tr>
<td>$J_{lc}$ [kN m$^{-1}$]</td>
<td>2.76</td>
</tr>
<tr>
<td>$J_{lle}$ [kN m$^{-1}$]</td>
<td>9.07</td>
</tr>
</tbody>
</table>

$^a$) Extrapolated value

Similar to this work, experimental results of the Mode I peak stress and the Mode I fracture energy of the DB1044 adhesive, obtained by Carlberger et al. (2009), have been re-evaluated with respect of the temperature in Walander et al. (2012) using the same statistical methods as in this work. From the evaluations in Walander et al. (2012), including the evaluations in this work, it is concluded that for both SP498 and DB1044, the cohesive parameters elastic stiffnesses, peak stress and the fracture energy are decreasing with an increase in the temperature in both loading modes. One exception is the Mode I fracture energy for SP498 where a slight increase is noticed with respect to the temperature. This increase is about 15 % when comparing the highest with the lowest mean value and about 6 % when comparing the highest with the lowest temperature in Table 3. In Fig. 9, the predictions of the Mode I fracture energy using a linear- in comparison with a second order- regression analysis almost coincide. Both the linear and the second order regression analyses give $R^2$ values near zero indicating an independence of the temperature for SP498. This is also supported by $K < 9.49$ in the Kruskal-Wallis test at a 5 % level of significanc. It can thus be concluded that the Mode I fracture energy of SP498 is independent of temperature in the tested interval $-40 \leq T \leq 80^\circ C$. The average value for all Mode I fracture energies is for SP498 $\mu_{J_{lc}} = 2.88$ kN m$^{-1}$.

The influence of the temperature on the fracture energy is significant in Mode II. The highest average value of the Mode II fracture energy in Table 3 occurs at $T = -30^\circ C$ and is about 40 % larger than the lowest
value at \( T = 80^\circ\text{C} \). The decrease in Mode II fracture energy corresponds to the prediction by Chai (2004) but not with the results in Banea et al. (2012) since their results show that the Mode II fracture energy has a maximum at the temperature \( T = 0.88 T_\alpha \) which for SP498 is about \( 55^\circ\text{C} \). Despite the quite low \( R^2 \) value, the result of the second order regression analysis, presented in Table 5, well predicts the average values of the fracture energies presented in Table 3. The largest difference between the second order estimate and the average values of the Mode II fracture energy is \( 6\% \) and occurs at \( T = -10^\circ\text{C} \). Obviously, and also since \( K > 9.49 \) in the Kruskal-Wallis test, the Mode II fracture energy cannot be considered as independent of the temperature.

For SP498, the lowest peak peel stress, considering the averages in Table 3, is obtained at the highest temperature, \( T = 80^\circ\text{C} \), in both modes. The highest peak stresses are obtained at the lowest temperature for both modes. At the highest temperature, the peak stress corresponds to \( 30\% \) of the peak stress at \( T = -40^\circ\text{C} \) for Mode I and \( 20\% \) of the peak stress measured at \( T = -30^\circ\text{C} \) in Mode II. The decrease in the peak stress with an increase in the temperature agrees with the results in Carlberger et al. (2009) for Mode I and in Banea et al. (2012) for Mode II. The peak stresses for SP498 decrease with about the same estimated linear slope, \( \hat{\beta}_1 = -312 \text{ kPa } \text{°C}^{-1} \) in Mode I and \( \hat{\beta}_1 = -367 \text{ kPa } \text{°C}^{-1} \) in Mode II; both with a high coefficient of determination, \( R^2 \geq 0.87 \). The peak stress of the DB1044 adhesive is estimated to decrease with an estimated linear slope of \( \hat{\beta}_1 = -345 \text{ kPa } \text{°C}^{-1} \) with \( R^2 \geq 0.76 \). This implies that the peak stresses can be considered to decrease linearly with respect to the temperature. At room temperature, the results of the maximum shear stress and the Mode II fracture energy do not agree with earlier experimental result obtained by Walander (2009) or Marzi et al. (2011), cf. Table 3. A possible explanation of this is given in chapter 7.

Both the modules and the elastic stiffnesses are noticed to decrease with an increase in the temperature as seen in Fig. 7. The results correspond to the assumptions in e.g. Banea et al. (2012), for both loading modes. However, in Mode II, a minimum of \( K_s = 0.92 \text{ TN m}^{-3} \) corresponding to \( G = 2.76 \text{ GPa} \) is predicted by the second order regression at \( T = 66^\circ\text{C} \), cf. Fig. 7. For predicting the elastic stiffnesses, linear estimations are not suitable since the linear models get low \( R^2 \) values. Furthermore it predicts \( K_s \) to be negative, cf. Fig 7. This is not physical possible. Using a second order regression gives higher \( R^2 \) values, even though a high level of determination is not achieved. By comparing Tables 3 and 5 it is noticed that the largest difference between the estimated and the average values is \( 23\% \) and occurs at \( T = 20^\circ\text{C} \) for Mode I and \( 68\% \) at \( T = 50^\circ\text{C} \) for Mode II.

![Fig. 10 Elastic modules vs. temperature. Solid Line: Mean values of the measured effective Young’s modulus, \( \tilde{E} \). Dashed line: Evaluated results of Young’s modulus, \( E \)](image-url)
By knowing the mean values of $\bar{E}$ and $G$ at equal temperatures, Young’s modulus for the adhesive is obtained using Eq. 15 and the result is presented in Fig. 10. Since the lowest evaluated temperature for the DCB and ENF experiments are not equal, an evaluation of $E$ at temperatures lower than -10°C is not possible.

$$E(\bar{E},G) = \frac{G(3\bar{E} - 4G)}{\bar{E} - G} \equiv \frac{K_s(3Kn - 4Kn)}{Kn - Ks} t$$

(15)

The results of $E$ in Fig. 10 agree well with earlier results of $E = 1.7$ GPa for SP498 performed at room temperature. Similar to Eq. 15, by using $\bar{E}$ and $G$, the effects of the temperature on Poisson’s ratio can be studied. However the results of $\bar{E}$ and $G$ spread too much and since the equation for Poisson’s ratio is very sensitive for variations of these parameters, the outcome is unreasonable. Due to this, the results of Poisson’s ratio are not presented. Similarly, due to the spread in results of $\bar{E}$ and $G$, the resulting $E$ in Fig. 10 should be treated as an indication rather than a definite measure, despite a good agreement with earlier experimental results.

Figure 11 shows representative strain rate data for each temperature. For each temperature, a polynomial of grade six is adapted to all experimental data. These polynomials are shown in Fig. 11 up to the mean value of the maximum strain for each temperature. It is noted that the strain rate increases with an increasing strain. This is due to the softening behaviour of the adhesive. The behaviour is different in Mode I than in Mode II. All curves appear to be similar in Mode I while in Mode II there is a gradual change in shape with a changing temperature. This can be understood from the shape of the cohesive laws, cf. Fig. 6. For Mode I, all curves resemble linear softening for most of the deformation. However, in Mode II, the curves develops from almost linear softening at -30°C to almost constant stress at 80°C. It is also shown that the maximum strain increases with an increasing temperature for both loading modes.

**Fig. 11** Representative strain rate vs. strain for each temperature group. *Left: Mode I. Right: Mode II*

### 6. Verification and simplified model

The results of the evaluation in Table 3 and 5 are verified by finite element analyses. By modelling the DCB and the ENF specimens with implemented cohesive laws, the simulated force vs. load point deformation relation is compared to the experimentally measured relations. A good fit gives confidence in the evaluated cohesive law. However, some caution is advised. It is now well understood that “crack-like” geometries are often much more sensitive to the fracture energy than to the other parameters of the cohesive law. The analysis with Abaqus standard version 6-11, consists of two-dimensional beam elements (B21) for the adherends and two-dimensional cohesive elements (Coh2D4) representing the adhesive layer. Two-dimensional beam- and cohesive-elements have two translational degrees of freedom (DOF) per node. However, beam elements do also have one rotational DOF per node. This is not present in the cohesive element. To relate this non common DOF to deformations of the cohesive element, rigid connector
elements (Conn2D4) are used. This is necessary for the simulations of the ENF-experiments in order to obtain shear in the cohesive elements. The specimens are modelled according to the dimensions in Table 1. Each adherend is modelled using 500 elements for the DCB- and 2700 elements for the ENF-specimens. This gives element lengths of 0.32 mm for the DCB and 0.1 mm for the ENF specimens. The cohesive elements have the same element size as for the adherends and the sizes are small enough to sufficiently capture crack creation and propagation. In the analysis, the adherends have elastic-plastic material data according to a tensile test of the Uddeholm Rigor steel, cf. e.g. Walander (2009). Before the yield limit at \( \sigma_y = 570 \text{ MPa} \), the steel is modelled as linear elastic with Young’s modulus \( E_a = 190 \text{ GPa} \) and Poisson’s ratio 0.33. The plastic response is modelled with tabulated data from the tensile test. Furthermore, the material behaviour for the steel is assumed not to vary with respect to the temperature.

The loading of the cohesive layer in the DCB-specimen is in pure Mode I and in the ENF-specimen it is almost in pure Mode II. Thus, the uncoupled cohesive behaviour is used in all analyses. This means that the stress-deformation-relation in Mode I is independent of the stress-deformation-relation in Mode II. Two different models are used for the adhesive: a simplified bi-linear model and a shape mimicking damage model. These are described below.

A bi-linear model is the simplest cohesive model. For each mode, it is constructed by the three parameters: the elastic stiffness \( (K_n, K_s) \) the peak stress \( (\sigma_{\text{max}}, \tau_{\text{max}}) \), and the fracture energy \( (J_{Ic}, J_{IIc}) \), cf. Fig. 12. If the deformation is smaller than \( w_i \) or \( v_i \), the response is linearly elastic. For increasing deformation the response is linearly softening. Above \( w_c \) or \( v_c \) the stress is zero and a crack starts to propagate. Two bi-linear cohesive laws for each specimen and temperature group are used. The parameters of the cohesive laws are taken from the mean values in Table 3 and from the prediction from the second order regression analysis in Table 5.

A shape-mimicking damage law has the same shape as the measured stress-deformation law. This is accomplished with the cohesive law \( \sigma = (1 - D)K_iw \), where \( D \) denotes the damage. The damage is a non-decreasing parameter with the initial value zero and the maximum value unity. At \( D = 1 \), a crack has formed. With \( w \) denoting the maximum deformation during the loading history, \( D = 1 - \sigma(K_iw_m) \). A similar expression is used in Mode II. For each evaluated temperature and loading mode, a representative cohesive law is first established by a filtering and adaptions procedure using all \( J \) vs. \( w/v \) data. These representative cohesive laws are presented in Figs. 6 and 13. These laws are then used to give the evolution of \( D \) which is used as input data to the simulations.

Fig. 12 Bi-linear model with notation for peel and shear

A shape-mimicking damage law has the same shape as the measured stress-deformation law. This is accomplished with the cohesive law \( \sigma = (1 - D)K_iw \), where \( D \) denotes the damage. The damage is a non-decreasing parameter with the initial value zero and the maximum value unity. At \( D = 1 \), a crack has formed. With \( w \) denoting the maximum deformation during the loading history, \( D = 1 - \sigma(K_iw_m) \). A similar expression is used in Mode II. For each evaluated temperature and loading mode, a representative cohesive law is first established by a filtering and adaptions procedure using all \( J \) vs. \( w/v \) data. These representative cohesive laws are presented in Figs. 6 and 13. These laws are then used to give the evolution of \( D \) which is used as input data to the simulations.
Fig. 13 Representative cohesive laws for each temperature group. *Left:* Mode I. *Right:* Mode II

The resulting force-deformation relations are presented together with the experimental results in Fig. 5. It is noted that the simulation results agree well with the experimental results, thus giving confidence in the data. One exception is the results of the simulations with bi-linear cohesive laws in Mode II at $T = 80^\circ C$. These results show a relatively large difference in Fig. 5. This shows that the bi-linear cohesive law is too limited to represent the more rectangularly shaped cohesive law at this temperature, cf. Fig. 13. A similar exception is the simulation in Mode II at $T = 20^\circ C$ using a bi-linear law. Too high initial stiffness is achieved from the simulation, cf. Fig. 5. The used parameters in this simulation, obtained by second order regression analyses, do not differ much from the mean values in Table 3.

The differences between the bi-linear and the shape-mimicking results are in most cases small. This shows that the shape of the cohesive laws has a minor influence on the structural behaviour of the present DCB- and ENF-specimens. An advantage by using bi-linear cohesive laws with parameters from the second order regression curves is that these cohesive laws can represent any chosen temperature within the evaluated temperature span.

As noted in Fig. 7, the scatter in experimental results for the elastic properties is substantial. In order to shed some light on the reason for this, Eqs. 9 and 12 are used with simulated experiments. By this procedure we eliminate all influences of errors in measurements and the manufacture of specimens. It is noted that small variations in the relations of $\frac{F}{w}$ and $\frac{F}{\nu}$ give large influences on $K_n$ and $K_s$ using Eqs. 9 and 12. Thus, small errors in the force and deformation measurements will lead to large errors in $K_n$ and $K_s$. The reliability of Eqs 9 and 12 is therefore evaluated by use of the same FE-analyses as used for the DCB and the ENF simulations. By assigning given values of the elastic stiffness $K_n$ and $K_s$ in respective FE-model, the stiffness of the specimen, $\frac{F}{w}$ and $\frac{F}{\nu}$, is evaluated numerically. For 30 equally distributed elastic stiffnesses, the measured stiffnesses $\frac{F}{w}$ and $\frac{F}{\nu}$ are presented with solid lines in Fig. 14 for each mode respectively. These stiffnesses are used with Eq. 9 and Eq. 12 to determine the corresponding value of the elastic stiffness. The results are presented with crosses.
The presented method for measuring the elastic peel stiffness is shown to give good agreement with the nominal results for effective modules within the measured values of SP498. The method for predicting the elastic shear stiffness in an ENF specimen deviates somewhat from the nominal values. The origin of this difference is traceable to effects of the thickness of the adhesive layer. It is noted that all experimental results in Fig. 7 show $G$ smaller than about 5 GPa. Thus, comparing with the data in Fig. 14, we can conclude that $G$ is somewhat overestimated.

7. Conclusions and discussion

It is concluded that all evaluated cohesive parameters for SP498 are decreasing with an increase in the temperature for both loading modes. The only exception is the Mode I fracture energy which is shown to be independent of the temperature in the evaluated temperature span $-40 \leq T \leq 80°C$. Simulated experiments using measured cohesive parameters show good agreement with the experimental results. The exceptions show that a bi-linear cohesive law is oversimplified in some cases. This is however mostly shown at low loads. In the load cases studied here, a bi-linear cohesive law with parameters from a second order regression curve suffice to give a very good estimate of the behaviour of the specimens.

Furthermore, the methods to measure the elastic properties show some promising features. It can be concluded that the influence of measurement errors is large. This can be compensated for by increasing the accuracy and increasing the number of evaluated specimens.

The experimental results for SP498 show that $\tilde{E}, E$ and $G$ are decreasing with an increase in the temperature. This result coincides with general knowledge of adhesives.

All experiments in Mode II at the temperature $T = 80°C$ show adhesive fracture. An adhesive fracture gives a weaker bonding strength than the strength of the adhesive.

At room temperature, two independent studies of SP498, cf. Walander (2009) and Marzi et al. (2011), show the Mode II fracture energy and the Mode II peak stress to be about 30 % larger than what is obtained in this work. Both studies use the method presented in Eq. 5b with the same specimen dimensions as presented in Table 1. In Marzi et al. (2011) also an End-Shear Joint specimen and a larger ENF specimen with elastic adherends are used. The latter is evaluated using Eq. 6. The methods and labs report consistent results. However, a two times larger load point displacement rate is reported. By this, the strain rate is expected to be about twice the value reported here. A study of the BM1044 epoxy adhesive in Mode I loading, cf. Carlberger et al. (2009), shows that an increase in the strain rate increases the both the fracture
energy and the peak stress. It is however questionable if a twice as high strain rate in Mode II gives such large influence.

The results in this paper show that it is possible to measure all three cohesive parameters for each pure mode loading case by use of the two simple DCB- and ENF-test specimens. Even though the measures tend to spread in values, this can significantly reduce the cost for performing experiments. Also, by using second order regression analyses, a cohesive law for each loading mode that is valid for any temperature within the temperature span $−30 \leq T \leq 80°C$, is presented. These results are desirable for the automotive industry when performing experiments and simulations of adhesives that include effects of the temperature. However, the results in this work do only consider pure mode loadings and by this only un-coupled simulations can be performed. In a normal crash test, a mixed-mode loading of the adhesive is mostly present. In order to perform mixed-mode simulations, the mixed mode behaviour needs to be determined experimentally. Today there exist only a handful of studies of the mixed mode behaviour of adhesives, cf. e.g. Högberg et al. (2007).

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Paper B
An evaluation of the temperature dependence of cohesive properties for two structural epoxy adhesives

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Abstract.
 Cohesive modelling provides a more detailed understanding of the fracture properties of adhesive joints than provided by linear elastic fracture mechanics. A cohesive model is characterized by a stress-deformation relation of the adhesive layer. This relation can be measured experimentally. Two parameters of the stress-deformation relation are of special importance; the area under the curve, which equals the fracture energy, and the peak stress. The influence of temperature of these parameters is analysed experimentally and evaluated statistically for two structural epoxy adhesives in the span from of -40°C to +80°C. The adhesives are used by the automotive industry and a temperature span below the glass transition temperature is considered. The results show that that temperature has a modest influence on the adhesives Mode I fracture energy. For one of the adhesives, the fracture energy is independent of the temperature in the evaluated temperature span. In mode II, the influence of temperature is larger. The peak stresses decreases almost linearly with an increasing temperature in both loading cases and for both adhesives.

Introduction
 The automotive industries are striving to minimize the weight of their products in order to reduce the fuel consumption and thereby the emissions. In addition, the manufacturers are facing requirements to improve the crashworthiness. This is often at the expense of an increased weight. By using lightweight materials such as aluminium or composites in the body structure and combine these with tough material, e.g. steel, at impact zones, a more optimized solution can be obtained. Today, the majority of body structures consist of alloyed steel sheets that are joined by spot welds. A disadvantage with spot welds is the difficulty to join steel with aluminium alloys. This has put focus on modern crash resistant epoxy adhesives that enable joining of dissimilar materials. When using adhesives in a body structure it is in terms of crashworthiness required that the adhesive layers remain intact during a crash. This secures that bonded material deform in a predicted mode to dissipate the kinetic energy safely.

With cohesive modelling, a stress-deformation relation is used to characterize the strength of an adhesive layer. This is a constitutive relation on a structural length scale between the traction exerted on the interfaces of the adhesive to the adherends and the separation of the interfaces. The separation equals the deformation of the adhesive layer. In the sequel, this relation is denoted a stress-deformation law. Fig. 1 indicates peel and shear deformation. These are characterized by peel deformation, $w$, and peel stress, $\sigma$, and shear deformation, $v$, and shear stress, $\tau$. The success of this characterization is due to the high toughness of modern adhesives. With brittle adhesives, we can expect to have to model the details of the fields more accurately.
The strength of adhesively bonded multi-material build-up structures can be adequately predicted using cohesive modelling and the finite element method, cf. e.g. [1]. This modelling provides a more detailed understanding of the fracture properties of adhesive joints than can be achieved with fracture mechanics. Methods to measure the cohesive properties in Mode I, II and in mixed mode loading are summarized in [2]. Typical stress-deformation relations are shown in Fig. 2. Two parameters of these relations are of special importance; the area under each curve which equals the fracture energy and the peak stress.

An automotive body structure is required to fulfil its requirements at all working temperatures. The relevant areas of a car body for which adhesives are of interest normally suffers the temperature range $-40^\circ\text{C} \leq T \leq 80^\circ\text{C}$. Some studies of the influence of temperature have been performed. In [3] it is shown that the stress-deformation relation for the epoxy adhesive DOW Betamate XW 1044-3 (DB1044) is strongly temperature dependent in Mode I. In this study, the entire stress-deformation law is evaluated at seven equally distributed temperatures with ten repeated experiments at each temperature. In [4] it is shown that the fracture energy for an structural epoxy adhesive decreases in the temperature region $0.7 < T / T_g < 1.0$, where $T_g$ denotes the glass transition temperature. For most epoxies it is about $100^\circ\text{C}$. Furthermore, it is shown that the yield strength decreases with increasing temperature and increases with increasing strain rate. In [4], the fracture energy is determined using an unstable specimen and the experiments are evaluated using linear elastic fracture mechanics (LEFM). That is, the entire cohesive relation is not captured.

Cohesive models are implemented in finite element software to simulate the behaviour of adhesively joined structures. To perform these analyses considering temperature, the temperature dependence of the adhesive layer has to be taken into account. The previous studies do not provide all the necessary data and therefore a cohesive model cannot yet be established. This implies that new experiments need to be performed. An adhesive that is of current interest by the automotive industry is the crash resistant epoxy SikaPower498 (SP498). In this work, temperature studies are performed on this adhesive in Mode I and Mode II. Statistical methods are used to evaluate the influence of temperature on the two important parameters, peak stress and fracture energy. Moreover, the results in [3] are re-evaluated using statistical methods.

**Methods**

The two most frequently used test specimens to measure Mode I and Mode II fracture properties for adhesives are the double cantilever beam (DCB) and the end notched flexure specimen (ENF) cf. Figs. 3 and 4, respectively. These specimens are used in the studies in [3] and [4]. The specimens
each consist of two adherends that are partially joined by an adhesive layer. The part of the specimens that is not joined by an adhesive layer is considered as a crack, and the start of the adhesive layer is denoted the crack tip.

For the DCB specimen the adherends are separated by a prescribed deformation \( \Delta \) and the reaction force, \( F \), is measured. The stress-deformation relation for the DCB specimen is given by Eq. 1 in which \( J \) is derived either by using beam theory, cf. [5], or by using the path independent \( J \)-integral, cf. [6].

\[
\sigma(w) = \frac{dJ}{dw} \equiv \frac{d}{dw} \left( \frac{2F \sin \theta}{b} \right),
\]

where, \( \theta \) is the rotation of the loading points and \( b \) is the specimen width.

A properly designed ENF-specimen gives almost a state of pure shear at the crack tip. A recently developed method to measure \( J \) for the ENF specimen is presented in [7] and is validated showing good agreement to input data using finite element analysis in [8]. From this method the stress-deformation relation of the adhesive layer is given as

\[
\tau(v) = \frac{dJ}{dv} = \frac{d}{dv} \left[ \frac{F}{b} \left[ (1-\eta)\sin \theta_1 - \sin \theta_2 + \eta \sin \theta_3 \right] \right]
\]
where $F$ denotes the load, $\eta$ is the distance between the left support and the loading point, $b$ is the specimen width and $\theta_1$, $\theta_2$ and $\theta_3$ are the rotations at the three supports. These are considered positive when increasing clockwise, cf. Fig. 4. Eqs. 1 and 2 provide the entire stress-deformation relation of the adhesive layer for each mode, respectively. In neither of these methods the constitutive properties of the adherends need to be known. Furthermore, both methods allow for plastic deformation of the adherends as long as no unloading from a plastic state takes place. Unloading from a plastic state would invalidate the path-independence of the $J$-integral used to derive Eqs. 1 and 2.

Regression analysis is used to evaluate the temperature dependence of the peak stress and the fracture energy; collectively denoted response variables, $y$. Simple models for the influence of the explanatory variable $x_1$ are given by a linear and a second order model, i.e.

$$y = x_1 \beta_1 + \beta_0 + \varepsilon.$$  
$$y = x_1^2 \beta_2 + x_1 \beta_1 + \beta_0 + \varepsilon. \quad (3a, b)$$

In this study, the only explanatory variable is the temperature; $\varepsilon$ is often denoted the disturbance term. The parameters $\beta_2$, $\beta_1$ and $\beta_0$ are to be estimated using the least square method. A major assumption in this method is that the explanatory variable is measured without errors. That is, the temperature is assumed to be measured exactly. If the influence of both $|\beta_2|$ and $|\beta_1|$ is small, the response variable is considered independent of the explanatory variable. The coefficient of determination $R^2$ is often used to indicate if the fit is good; if $R^2$ equals 1, all data points are on the fitted curve. On the other hand, if there is no dependence of the response variable on the explanatory variable, $R^2$ equals zero cf. e.g. [9].

Another way of analysing dependence, not assuming a normal distribution, is to use a non-parametric rank test such as the Kruskal-Wallis test, cf. [10]. This one-way analysis of variance of ranks enables testing several populations against each other. If the number of samples of each population is large enough, a certain test variable $K$ can be assumed to be chi-square, $\chi^2$-distributed. If $K < \chi^2_{g-1}$ the medians of each population are assumed equal. The value $\chi^2_{g-1}$ is determined to give a certain probability. Thus, indicating that there is no dependence of the explanatory variable. The procedure and notation is described below. All observations are first ranked independent of the temperature. That is, the smallest value is given the rank 1; the second smallest is given the rank 2 and so on until the largest value is given the rank $n$ equal to the total number of observations. All observations are then grouped in samples corresponding to the temperature. This gives $g$ samples corresponding to the $g$ temperatures. In each group, the average rank of the observations in each group is calculated, $\tilde{r}_i$, where $i = 1, 2, \ldots, g$. With $n_i$ equal to the number of observations in sample $i$, the test variable $K$ is calculated according to

$$K = \frac{12}{n(n+1)} \sum_{i=1}^{g} n_i \tilde{r}_i^2 - 3(n+1) \quad (4)$$

Using a table of the $\chi^2_{g-1}$ distribution, the probability that the medians are equal is estimated. More formally, the null hypothesis that there is no difference in the response variable between the samples is rejected if $K \geq \chi^2_{g-1}$. With a 5% risk of rejecting the null hypothesis even if it is true, we need $K$.
smaller than 9.49 with five evaluated temperatures; with seven evaluated temperature we need $K$ smaller than 12.59. This risk is denoted the level of significance.

**Experiments**

Two experimental set-ups for the performed experiments are shown in Fig. 5. Here, the climate chambers are not shown. With the DCB testing machine, two crossheads are separating the load points on both sides with a prescribed loading rate, $\Delta = 10 \mu m/s$. This is slow enough to consider the test quasi-static. In these experiments, the crack tip separation, $w$, as well as the load point displacement, $\Delta$, are measured using linear variable differential transformers (LVDT). The rotation of the loading point, $\theta$, is measured using an incremental shaft encoder and the load, $F$, is measured using a load cell. Only the rotation of one of the adherends is measured and it is assumed that the specimen deform symmetrically. This assumption has been tested in earlier experiments and found to give sufficient accuracy. An external climate chamber is used for the temperature span $-40^\circ C \leq T \leq 80^\circ C$. The experiments are performance identically as in [3] with exception for the number of evaluated temperature intervals.

![Experimental set-ups with deformed specimens.](image)

For the ENF set-up, a servo hydraulic test machine is used. The load acts at $\eta = 0.7$, cf. Fig. 4 and 5. Two LVDTs for measurements of each rotations, $\theta_i$, and one LVDT for measuring the shear deformation, $v$, are used. The load point displacement, $\Delta$, is also here given a prescribed loading rate, $\Delta = 3.8 \mu m/s$, slow enough to be considered the test as quasi-static. The aim is to test the adhesive at the same temperatures as in the DCB-experiments. However, the climate chamber has a lower limit of $-30^\circ C$. Thus, $-30^\circ C$ is the lowest evaluated temperature for the ENF experiments.

The adherends of the specimens are made of the Uddeholm Rigor tooling steel that, according to tensile tests, allows for engineering strains up to 14 % before plastic straining start, cf. [8]. The thickness of the adhesive layer is nominally $t = 0.3$ mm for all specimens with SP498 and $t = 0.2$ mm for the DB1044 adhesive. The DCB specimens has the dimensions in mm, $L = 160$, $a = 80$, $h = 6.5$ and $b = 5$, cf. Fig. 3. The ENF specimens has the dimensions in mm $L = 200$, $a = 70$, $h = 10$ and $b = 10$, cf. Fig. 4. In order for the $J$-integral to be valid the stresses in the adhesive layer at the right end of the specimen, cf. Fig. 4, needs to be negligible. Therefore an overhang of 50 mm is used for the ENF specimens, cf. Fig. 5. Also in order to ensure that the ENF specimen remain on the support during deformation, an overhang of 20 mm is used on the left end. This gives the total specimen length of 270 mm.
Evaluation and results
From the experiments, stress-deformation relations like the ones in Fig. 2 are obtained using Eqs. 1 and 2. For each experiment, the two parameters fracture energy, $J_c$, and the peak stress, are presented in Fig. 6. The results of the DB1044 adhesive in [3] are also included in this evaluation. The DB1044 adhesive have significant lower fracture energies than the SP498 adhesive. During manufacturing of the specimens, air bubbles may arise in the adhesive layer. In order to justly be able to compare two different adhesives, all specimens are carefully investigated after the experiments. If an adhesive layer contains air bubbles in the region close to the crack tip, it is excluded from the evaluation since it affects the measured fracture energy.

From Fig. 6 it is concluded that the peak stresses decrease with an increase in temperature for both adhesives, both in Mode I and in Mode II. Regarding the fracture energy, indications that the fracture energy is independent of temperature in Mode I are given in Fig. 6. This result is however not observed in Mode II where the fracture energy clearly decreases at temperatures above 50°C which corresponds with the observation in [4].

The dashed and the dashed-dotted lines show the results of a first and a second order regression analysis, respectively.

By performing regression analyses, the estimated trend with respect to the temperature are calculated by the least square method, cf. Fig. 6. The estimated coefficients of regression in Eq. 3a and Eq. 3b are presented in Table 1 where the temperature in centigrade has been used.

**Mode I.** The top graphs in Fig. 6 show the results from Mode I loading. As shown, the first and second order regression curves virtually coincide for the fracture energy of both adhesives. For SP498 $R^2$ is very small indicating that there is no dependence of the temperature on the fracture energy. This is supported by $K = 6.04$ in the Kruskal-Wallis test at the 5 % level of significance. Thus, it is safe to state that the fracture energy in Mode I does not depend on the temperature in the tested interval. For DB1044, there is a slight influence of the temperature. We get $K = 31.2$ and would have needed a value below 12.59 with the present level of significance. The mean values of the Mode I fracture energies are $\mu_{SP498} = 2.68 \text{ kN m}^{-1}$ and $\mu_{DB1044} = 825 \text{ N m}^{-1}$ for the SP498 and the DB1044 adhesive, respectively.

The peak stresses decreases with increasing temperature. For SP498, a linear regression curve gives a good fit; for DB1044 a parable fit the data accurately, both with $R^2$ values near 0.9.

**Mode II.** The bottom graphs in Fig. 6 shows the results from Mode II loading for SP498. The fracture energy as well as the peak stress cannot be considered as temperature independent. The Kruskal-Wallis test gives $K = 10.3$ and a value below 9.49 would be necessary with the 5 % level of significance. The second order regression analysis predicts a continuous decrease in fracture energy with respect to the temperature. However, with a small $R^2$ value (0.35). Both a linear and a second order regression curve give high $R^2$ values for the peak stress.

Table 1. Estimated coefficients of regression and test value, $K$.

<table>
<thead>
<tr>
<th>Mode</th>
<th>Peak stress</th>
<th>Fracture energy, $J_c$</th>
<th>$\hat{\beta}_2$</th>
<th>$\hat{\beta}_1$</th>
<th>$\hat{\beta}_0$</th>
<th>$R^2$</th>
<th>$K$</th>
<th>$\hat{\beta}_2$</th>
<th>$\hat{\beta}_1$</th>
<th>$\hat{\beta}_0$</th>
<th>$R^2$</th>
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<tr>
<td></td>
<td>[kPa °C$^{-2}$]</td>
<td>[kPa °C$^{-1}$]</td>
<td>[MPa]</td>
<td>[-]</td>
<td>[mN m$^{-1}$ °C$^{-2}$]</td>
<td>[N m$^{-1}$ °C$^{-1}$]</td>
<td>[kN m$^{-1}$]</td>
<td>[-]</td>
<td></td>
<td></td>
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<tr>
<td>DB1044</td>
<td>3.19</td>
<td>-491</td>
<td>32.0</td>
<td>0.85</td>
<td>-25.3</td>
<td>0.37</td>
<td>0.88</td>
<td>0.55</td>
<td>31.2</td>
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<td></td>
<td>-</td>
<td>-345</td>
<td>34.3</td>
<td>0.76</td>
<td>-</td>
<td>-1.53</td>
<td>0.86</td>
<td>0.39</td>
<td>-</td>
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<td>SP498</td>
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<td>-270</td>
<td>50.6</td>
<td>0.88</td>
<td>-1.91</td>
<td>1.28</td>
<td>2.93</td>
<td>0.05</td>
<td>6.04</td>
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<td>-</td>
<td>-312</td>
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<td>0.87</td>
<td>-</td>
<td>1.21</td>
<td>2.93</td>
<td>0.05</td>
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<td>-426</td>
<td>38.1</td>
<td>0.82</td>
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<td>-22.8</td>
<td>9.62</td>
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<td>10.3</td>
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<td>-</td>
<td>-325</td>
<td>40.5</td>
<td>0.78</td>
<td>-</td>
<td>-27.4</td>
<td>9.50</td>
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<tr>
<td></td>
<td>a) Second order regression analysis</td>
<td>b) Linear regression analysis</td>
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</table>

It is also interesting to note that the linear models for all peak stresses have about the same slope, i.e. $\hat{\beta}_1$ varies from about -310 to -350 kPa °C$^{-1}$.

**Conclusions**

Within the evaluated temperature interval, $-40 \leq T \leq 80$ °C, the evaluation show a temperature independent Mode I fracture energy for SP498. For DB1044, there is a small influence of the
temperature in Mode I. However, the influence is so small that it is reasonable to ignore the influence for engineering purposes. There is an influence of the temperature on the fracture energy in Mode II with a decrease in fracture energy with an increase in temperatures. The peak stresses decreases almost linearly with increasing temperature for both epoxy adhesives and for both Mode I and II for SP498.

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References