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Research Article

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Evaluation of Critical SERR in the Presence of Heterogeneous Interfaces in Bonded Joints: Application to Parallel Defects in DCB Titanium alloy laser treated specimens

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Abstract:

In this study, longitudinal defects are examined in the context of adhesively bonded structures, a subject that has remained understudied in previous investigations. Employing fracture mode I loading on Double Cantilever Beam (DCB) specimens with variable adhesion strength, we investigate the influence of weakened adhesion areas on the critical Stress Energy Release Rate (SERR). Modifications of titanium alloy specimen surfaces, with nanosecond laser treatment, alter their surface morphology dynamically. Our mechanical testing unveils a pronounced effect of weakened adhesion areas on SERR, necessitating a nuanced understanding.

A comprehensive model is presented that takes both transversal and longitudinal defects into account, providing detailed insight into the effect of finite defects on SERR values. The theoretical models meticulously calculate overall critical SERR ($G_c$) in heterogeneous bond lines, exhibiting a compelling correlation with experimental results. The study underscores the non-linear variation of critical SERR ($G_c$) with the fraction of strong adhesion area, a phenomenon influenced by the crossover between areas of good and weakened adhesion.

Keywords: DCB testing, laser texturizing, moment-based approach, crack front shape, crack propagation, fracture energy, longitudinal defects, mechanical resistance, adhesive stiffness.

1. Introduction:

Adhesive bonding has been extensively used in various industrial applications for several decades, due to its numerous advantages such as weight reduction, homogeneous stress distribution, and enhanced durability compared to conventional methods like bolting or riveting [1]. Nonetheless, the manufacturing of adhesively bonded joints remains a critical process, demanding meticulous
attention to prevent the inclusion of defects within the bond line. It seems challenging to prevent the presence of defects during various bonding operations, especially when treating surfaces.

Whether adhesive or cohesive, these defects cause the assembly to fail. Based on where the defect appears, it can be distinguished from other types of failures. A cohesive defect leads to cohesive failure, which means that the crack spreads within the adhesive. On the other hand, an adhesive defect causes adhesive failure, which means that the crack spreads along the adhesive-substrate interface. There can also be a third type of failure which is a combination of both adhesive and cohesive failure [2][3][4][5]. These defects affect the mechanical resistance of the bonded joint measured by calculating the critical Strain Energy Release Rate SERR, $G_c$, or its toughness which is generally determined using standard mechanical characterization tests like ASTM D5528-01 or ASTM D6671. These tests realized on bonded joints are inspired by DCB tests, originally developed by Mostovoy and Rippling [6]. Various experimental arrangements and test geometries have been proposed since then for measuring interface toughness in modes I, II, III, or mixed modes [7][8].

The impact of interfacial flaws on joint resistance has been studied for many years focusing on studying the macroscopic effect of substrate surface flaws by calculating the total fracture energy (which is the critical strain energy release rate, $G_c$) of the bonded assembly based on local adhesion properties of the flaws depending on their size and location [9][10][11][12][13].

The types of defects treated before in references cited above and in [14], [15], [16] and [17] are distributed across the entire width (transversal defects) or along the entire length (parallel defects: crack propagation is parallel to the defect length along the substrate), Fig. 1. And it is necessary to mention that not much interest has been given to parallel defects, despite their significance. It is crucial to consider them, as they represent an obligatory step in estimating the critical SERR of finite defects (finite width and finite length) within the bonded area.
In practice, interfacial flaws exhibit random shapes, and for simplification reasons, we must consider a well-defined, ideal shape that can closely approximate the original flaw's shape with accuracy. The decomposition of the finite defect into transversal with finite length and parallel with finite width results in an overlap between the two shapes. Researchers have developed a mathematical model for crack growth based on the effects of transversal defects \([17] [18]\). The second part, which is addressed in this research paper, is dedicated to investigating the impact of parallel defects on the total critical SERR \(G_c\) of the specimen containing these defects. Subsequently, a comprehensive model will be proposed to combine the effects of both transversal and parallel defects, providing an accurate estimation of specimens’ fracture energy.

2. Materials and methods

The experimental work in this study was performed under fracture mode I loading conditions on adhesively bonded DCB specimens (see Fig. 2) to investigate the interaction between the crack and a flaw. A flaw is characterized by low adhesion, and it is referred to as a “weak” interface, in contrast to a properly treated surface with good adhesion, which is termed a “strong” interface.

2.1. Materials and surface preparation before bonding

DCB specimens, Fig. 2, are composed of two Titanium alloy Ti6Al4V substrates. This material is widely used in many applications such as aeronautics and this is due to its high mechanical characteristics, low density, and excellent corrosion resistance. Substrates dimensions are Length \(L = 200\text{mm}\), Width \(w = 25\text{ mm}\), and thickness \(t = 1.6\text{ mm}\). The Young’s modulus of Titanium alloy Ti6A14 is about 110 GPa;
To modify the surface morphology to make it suitable for bonding, a nanosecond laser treatment in an ambient atmosphere was performed. Laser techniques have been developed as an alternative solution to traditional machining and chemical treatments sometimes harmful to human beings and the environment [19] [20].

Before the laser surface treatment, the substrates were thoroughly degreased with acetone in an ultrasonic bath. Then, substrate surfaces were irradiated with a 4W laser, swept at a speed of 250mm/s in both longitudinal and transversal directions across the substrate, with a step size of 10 µm. A Boras G30 laser system (Eolite Systems) was employed, generating a visible beam with a wavelength of 550 nm. After laser treatment, substrates were cleaned in a deionized water ultrasonic bath and then dried with pure hot air. This treatment led to a mechanically active surface by creating significant roughness, see Fig. 3, which improves the mechanical anchoring of the adhesive, as shown in Fig. 4. In addition, contact angle measurements using distilled water (not presented here but visible in [21] indicate complete wetting, signifying a high surface energy suitable for bonding.

![Fig. 2 DCB specimens’ configuration](image)

Fig. 2 DCB specimens’ configuration

![Fig. 3 Surface aspect after laser treatment on titanium alloy substrates (Power 4W, scanning speed 25 mm/s, step size 10 µm).](image)

Fig. 3 Surface aspect after laser treatment on titanium alloy substrates (Power 4W, scanning speed 25 mm/s, step size 10 µm).
Titanium alloy substrates are joined using an epoxy-based unsupported adhesive film. To ensure uniform thickness across the length and width of the bonded area and to confine the adhesive during the curing process, a 0.1mm PTFE sticker film was bonded to the perimeter, as illustrated in Fig. 5. Additionally, a pre-existing crack with a length of \(a_0\) was introduced into the DCB specimens to initiate crack propagation.

Laser-treated surfaces are bonded together to create a strong adhesion area. However, in the case of a weak adhesion area, two surfaces are bonded, one of which has been treated (TiA) as illustrated in Fig. 6. The initial step of this study involves determining the critical energy strain rates for both weak and strong adhesion areas. Then, to modify the width of the strong adhesion area, a triangle-shaped pattern (TiT) has been created by laser treatment, see Fig. 6, modifying then the parameter \(d\). This triangle shape has also been created in a reverse direction (TiTR). These configurations will allow us to understand how the critical SERR varies continuously with crack length. This understanding will help validate the analytical model that we will develop in this study.
The test setup is presented in Fig. 7. Specimens are loaded with a dual actuator system composed of two EZ001 Zwick electromechanical actuators. The actuators are placed horizontally opposite each other. Once attached to the end block of the machine, the specimen is vertical. A constant displacement rate $\dot{\Delta}=1\text{mm/min}$ is set on both actuators resulting in a $2\text{mm/min}$ specimen opening rate.

3. Calculation of the critical SERR $G_c$

To calculate the SERR $G_c$, it is necessary to determine the specimen compliance $C$. Several assumptions must be made for this purpose. We assume that the interface (substrate/adhesive/substrate) is infinitely stiff, and the substrates are modeled as Euler-Bernoulli flexible beams [16] [17] [22]. Based on Fig. 2, we can then write the compliance $C$ as follows:
\[ C = \frac{2\Delta}{P} = \frac{2a^3}{3EI} \quad (1) \]

With \( P \) is the applied force by the two tensile actuators on the specimen, \( 2\Delta \) is the opening displacement of the specimen measured on the loading line, \( a \) is the crack length measured between the crack tip and the loading line, \( E \) is Young’s modulus of the substrates, \( I = \frac{wt^3}{12} \) is the quadratic moment of the cross-section of the substrate with \( w \) and \( t \) are the width and thickness of the substrates respectively.

The compliance \( C \) is used to express SERR \( G \), as follows:

\[ G = \frac{1}{2w} \left( \frac{dC}{da} \right) P^2 = \frac{a^2P^2}{wEI} \quad (2) \]

Moreover, it is possible to write the instantaneous crack length, determined from specimen compliance in equation (1) as:

\[ a = \sqrt{\frac{3EI \Delta}{2P}} \quad (3) \]

The crack propagation condition is reached when \( G = G_c \), \( G_c \) being the critical SERR. Assuming a uniform bond line, \( G_c \) can be considered to be constant along the crack propagation path. Removing the parameter \( a \) in equations (1) and (3), we find the expression describing the evolution of \( P(\Delta) \) during the crack propagation regime.

\[ P = \left( \frac{4}{9}EI \right)^{1/4} (wGc)^{3/4} \frac{1}{\sqrt{\Delta}} = \alpha \Delta^{-1/2} \quad (4) \]

Bonded surfaces of substrates contain as can be seen in Fig. 6, some areas with good adhesion and others with poor adhesion along the width. So, we can use a fraction that describes the area with good adhesion noted \( f_s \) and area with poor adhesion noted \( f_w \), see Fig. 8.

![Diagram of crack propagation](attachment:diagram.png)

**Fig. 8** Calculation of the fraction of strong adhesion zone

Then, the fraction of good adhesion, \( f_s \), is calculated as:
\[ f_s = \frac{d}{w} \]  
(5)

With \( d \) is the evolving width of the region with good adhesion and \( w \) is the width of the substrate.

The fraction that represents poor adhesion is calculated as:

\[ f_w = 1 - f_s = \frac{w - d}{w} \]  
(6)

The most intuitive approach to evaluating the apparent critical restitution rate during crack propagation in the presence of a longitudinal defect is to use a simple mixture law. We introduce then \( G_{cw} \) and \( G_{cs} \) as the intrinsic critical SERR of poor and good adhesion interfaces. These contributions are presumed to act in parallel, drawing an analogy with the effective elastic modulus of a unidirectional, long-fiber composite material, aligned in the fiber direction. Then, the overall critical SERR \( G_c \) is written as:

\[ G_c = f_s G_{cs} + (1 - f_s) G_{cw} \]  
(7)

The second approach consists of using shear forces at the crack front. The opening force, \( P \), applied to separate adherents must be balanced by the cohesive forces in the substrates at the crack front which can be decomposed as follows:

\[ P = P_s + P_w \]  
(8)

Where \( P_s \) is the local separation force in the area with strong adhesion, and \( P_w \) represents the local separation force applied in the region with poor adhesion. And using equation (4), we can write:

\[ P = \left( \frac{Ewt^3}{27\Delta^2} \right)^{1/4} \left[ w_s G_{cs}^{3/4} + d \ G_{cw}^{3/4} \right] \]  
(9)

Equation (4) can be transformed into:

\[ G_c = \frac{3P^{4/3}}{t} \left( \frac{\Delta^2}{Ew^4} \right)^{1/3} \]  
(10)

And replacing \( P \) of equation (9) with equation (10), we can obtain equation (11) which represents the second approach for calculating the overall critical SERR \( G_c \) in a heterogeneous bond line:

\[ G_c = G_{cs} \left[ r^{3 \frac{3}{4}} + f_s \left( 1 - r^{3 \frac{3}{4}} \right)^{\frac{4}{7}} \right] \]  
(11)

With \( r = \frac{G_{cw}}{G_{cs}} \).
The third theory suggests that crack propagation is managed by bending moments at the crack front. From equation (2), the total bending moment is expressed in function of the critical SERR $G_c$ as:

$$M = a.P = \sqrt{w\, E\, I\, G_c} = \frac{w}{2} \sqrt{\frac{E t^3}{3}} \sqrt{G_c}$$

(12)

Considering a parallel distribution of the moments at the crack front, the total bending moment can be written as:

$$M = M_s + M_w$$

(13)

With:

$$M_s = \frac{d}{2} \sqrt{\frac{E t^3}{3}} \sqrt{G_{cs}}$$

(14)

$$M_w = \frac{w - d}{2} \sqrt{\frac{E t^3}{3}} \sqrt{G_{cw}}$$

(15)

Then to determine $G_c$ from the total moment $M$ from equation (13), we can write using $r = \frac{G_{cw}}{G_{cs}}$:

$$G_c = G_{cs} \left( f_s + (1 - f_s) \sqrt{r} \right)^2$$

(16)

This theoretical model presents three methods, equations (7), (11), and (16) to calculate the total critical SERR $G_c$, of DCB specimens containing surfaces presenting poor and good adhesion. This last method, described in equation (16), is a bit similar to that presented in [15]. This model has been tested using a 4mm thick flexible polycarbonate plate bonded to a rigid aluminium block using an epoxy adhesive. It has been found that, in this configuration of materials, the correspondence between experimental results and those given by the model does not match very well. In the same reference, the authors proposed an additional parameter that adjusts values of critical SERR $G_c$, corresponding to fractions of good adhesion zones excluding $f_s=0$ and $f_s=1$ where the crossover is absent. This adjustment was proposed to account for reductions in effective widths, over which local fracture energies are invalid.

4. Mechanical Testing and Results

This section begins by studying homogenous interfaces to calculate the critical SERR for an interface with good adhesion $G_{cs}$ and the critical SERR for an interface with poor adhesion $G_{cw}$. Those two parameters are necessary to calculate the energies contrast $r = \frac{G_{cw}}{G_{cs}}$.

4.1. Characterizing homogeneous interfaces
To calculate the critical SERR of interfaces with good and poor adhesion, experimental cracking tests on the DCB specimens TiS+TiS and TiS+TiA were used (see section 2). **Fig. 9** shows the variation of the load P in function of the opening displacement Δ.

![Fig. 9](image.png)

**Fig. 9** Force, P, versus opening displacement, Δ, during DCB experiment of TiS+TiS, TiS+TiA specimens

The fracture interfaces of the initial section exhibit an adhesive nature, indicative of poor adhesion, as in **Fig. 10**. Consequently, the critical SERR $G_{c_w}$ for an interface with poor adhesion can be calculated. Instead, in the remaining substrate, the fracture is cohesive, which indicates a good adhesive region. Then, the critical SERR $G_{c_s}$ for a good interface can be determined.

The first part of the square and circle-marked curves represents crack propagation within an interface presenting poor adhesion. Then, when the crack reaches an interface with good adhesion, a reload happens to reach the load level corresponding to the propagation in an interface with a good adhesion described by the square-marked curve.

![Fig. 10](image.png)

**Fig. 10** fracture of interfaces in regions with poor and good adhesion
Based on Fig. 9 and the TiS+TiT curves, it seems that a stick and slip phenomenon occurred while crack propagation was occurring at a good adhesion interface. It is a mixture between crack arrest and crack jump, visible in Fig. 10, and this can be related to bond line stiffness, as explained by Guo et al [23]. The release of potential energy stored during crack initiation leads to a sudden and rapid propagation of the crack accompanied by a lower critical SERR $G_c$. Fig. 11 displays the evolution of the critical SERR in function of crack length for both interfaces with good and bad adhesion. For the reason of clarity, data reduction was applied to Fig. 11 by eliminating the stick-and-slip effect from the curves. Only the critical SERR $G_c$ at crack arrest was considered.

**Fig. 11** Strain energy release rate variation in function of crack length

The critical SERR of the interface with poor and good adhesion is equal to $G_{cw} = 300 \text{ J/m}^2$ and $G_{cs} = 1500 \text{ J/m}^2$ respectively. These values will be used in the next paragraph to calculate the total critical SERR $G_c$ of a heterogeneous interface.

### 4.2. Interfaces with longitudinal pattern

TiS+TiT and TiS+TiTR specimens bonded with a structural adhesive containing interfaces with both good and poor adhesion along the substrate’s width and oriented in the same direction as crack growth were tested as explained in Section 2. The interfaces of fracture are illustrated in Fig. 12; the load-displacement curves appear in Fig. 13.
Fig. 12  Fracture interfaces for TiS+TiT and TiS+TiTR specimens showing cohesive fracture in both cases.

An adhesive failure was observed in areas with poor adhesion, and a cohesive failure occurred in areas with strong adhesion for TiS+TiT specimens. Observation of adhesive residue on the surface of TiS+TiTR specimens indicated that the cohesive failure was not right at the middle of the adhesive, but very near the interface, as shown in Fig. 12.

Fig. 13  Mechanical behaviour of TiS+TiT Titanium alloy DCB configuration
Fig. 14  SERR evolution in function of crack length for both TiS1+TiT1 and TiS2+TiT2 and comparing with critical SERR of specimens having good and poor adhesion

Fig. 15  Mechanical behaviour of TiS+TiTR Titanium alloy DCB configuration

Fig. 16  SERR evolution in function of crack length for both TiS1+TiTR1 and TiS2+TiTR2 and comparing with critical SERR of specimens having good and poor adhesion
When the peak of the TiS+TiT curve occurs in Fig. 13, it corresponds to the minimum level of adhesion. The TiS+TiT sample behaves in this region similarly to a sample with totally low adhesion. As the crack propagates, the area of good adhesion gradually increases, which explains the increase in critical SERR along the substrate as illustrated in Fig. 14.

The TiTR specimens follow the same reasoning, Fig. 15. The crack initiates at an interface with good adhesion which corresponds to the critical strain energy release rate of the TiS specimen. Then, the interface with good adhesion decreases which leads to a decrease in the critical strain energy release rate as shown in Fig. 16.

Equations (11) and (16) were developed to estimate the total critical SERR $G_c$, which represents the combination of $G_{c_s}$ and $G_{c_w}$ controlled by fractions $f_s$ or $f_w$, following the triangular geometry of the bonded area. The triangular regions in Fig. 17 and Fig. 18 exhibit strong adhesion indicated by a critical SERR $G_{c_s}$, while the remaining bonded area has weak adhesion marked by a critical SERR $G_{c_w}$. The first phase consists of determining the fraction associated with the interface displaying good adhesion for both TiS+TiT and TiS+TiTR configurations.

Fig. 17 Calculation of the fraction of the interface with good adhesion for TiT configuration

![Graph of Fig. 17](image)

$$d = 0.075a - 2$$

Fig. 18 Calculation of the fraction of the interface with good adhesion for TiTR configuration

![Graph of Fig. 18](image)

$$d = 0.046a + 12.571$$

The fractions of the interface with good adhesion in TiT and TiTR configurations are calculated using equation (5) and Fig. 17 and Fig. 18 respectively as follows:

$$f_{s_{TIT}} = \frac{2d}{w} = \frac{0.15a - 4}{w} \quad (17)$$

$$f_{s_{TITR}} = \frac{2d}{w} = \frac{0.129a - 25.14}{w} \quad (18)$$
For TiT configuration, we replace equation (17) in equation (11) and equation (18) in equation (11) and we obtain respectively:

\[
G_{c_{TiT}} = G_{cs} \left[ r^{3/4} + \frac{1.5a - 4}{w} (1 - r^{3/4}) \right]^{4/3}
\]  (19)

\[
G_{c_{TiTR}} = G_{cs} \left[ r^{3/4} + \frac{0.129a - 25.14}{w} (1 - r^{3/4}) \right]^{4/3}
\]  (20)

Also, for TiTR configurations and using the same equations (16) with (17), and (16) with (18) we obtain respectively:

\[
G_{c_{TiT}} = G_{cs} \left( \frac{0.15a - 4}{w} + \left( 1 - \frac{0.15a - 4}{w} \right) \sqrt{r} \right)^2
\]  (21)

\[
G_{c_{TiTR}} = G_{cs} \left( \frac{0.046a + 12,571}{w} + \left( 1 - \frac{0.046a + 12,571}{w} \right) \sqrt{r} \right)^2
\]  (22)

Fig. 19  Critical SERR \(G_c\) variation in function of fraction of strong adhesion area and approximation using equations (19) and (21) for TiS+TiT specimens
**Fig. 20** Critical SERR $G_c$ variation in function of fraction of strong adhesion area and approximation using equations (20) and (22) for TiS+TiTR specimens

The direct approach to estimating the critical SERR $G_c$ involves using the equation (7), which is graphically represented by the simple mixture law. However, this law demonstrates linear evolution and deviates significantly from the evolution determined through experiments that is visibly curved.

As the fraction of good adhesion area varies continuously in specimens of TiS+TiT and TiS+TiTR, the critical SERR $G_c$ should also vary smoothly and continuously. In **Fig. 19**, it is evident that the relationship between the critical SERR $G_c$ and the fraction $f_s$ is non-linear and exhibits slight convexity. This same behaviour is also observed in **Fig. 20**, which illustrates the evolution of the critical SERR $G_c$ for TiS+TiTR specimens.

The weak region in proximity to strong adhesion zone affects strong adhesion and decreases the critical SERR $G_c$. This decline in critical SERR $G_c$ is due to the increase in local stress concentration when transitioning from an adhesive fracture in the weak adhesion zone to a cohesive fracture in the strong adhesion zone.

Based on equation (1), which is adapted for a triangular strong adhesion zone shape in this case and described by equation (19), the analytical model is not very accurate in modeling the variation in critical SERR $G_c$ for both TiS+TiT and TiS+TiTR specimens. For almost all critical SERR $G_c$ values, the standard deviation described by the error bars is large. It can be used, however, if the estimation errors it may cause are considered. The model which is more convenient to describe the real variation of the critical SERR $G_c$ in function of the good adhesion zone fraction is equation (16) based on the addition of moments at crack fronts in zones with weak and good adhesion ($\approx 4\%$ of the mean standard deviation). The values of the critical SERR $G_c$ corresponding to the fractions, excluding $f_s=0$ and $f_s=1$ (because the crossover is absent), cause the non-linearity of the variation,
and this is due to the crossover between the good adhesion and weak adhesion zones as shown in Fig. 21. The width of crossover is related to adhesive toughness. The tougher the adhesive, the smaller the crossover, resulting in a higher critical SERR $G_c$. The correlation between the analytical approximation using equation (16) and the experimental results is satisfactory, as a very good match was obtained without the need for correction or adjustment of parameters due to crossover width.

**Fig. 21** Crossover between adhesive fracture caused by weak adhesion and cohesive fracture

**Conclusion**

This study focused on the impact of interfacial flaws on joint resistance, particularly longitudinal defects, which had not been extensively studied in previous research. The experimental work involved fracture mode I loading conditions on adhesively bonded DCB specimens with weak and strong adhesion areas. Surface treatments, including nanosecond laser treatment, were applied to modify surface morphology and improve mechanical anchoring of the adhesive.

Mechanical testing revealed that longitudinal defects, in the form of weak adhesion areas, significantly influenced the critical SERR $G_c$ and crack growth in the bonded joints. The study proposed a comprehensive model that combined the effects of both transversal and longitudinal defects, providing a more accurate description of the influence of localized finite defects on SERR values. Theoretical models were developed to calculate the overall critical SERR $G_c$ in heterogeneous bond lines, considering the presence of both good and poor adhesion areas. A satisfactory correlation was found between experimental results and theoretical predictions without additional correction parameters. The study highlighted the non-linear variation of critical SERR $G_c$ with the fraction of the strong adhesion area, influenced by the crossover between good and weak adhesion zones.

The analytical model described in Equation (16) is conservative. However, it's important to note that the calculation doesn't fully account for adhesive toughness due to the simplified assumption of
simple beam theory. This theory assumes that the bond line is infinitely tough, and only the substrates’ rigidity is considered in the calculation. Therefore, while the model provides a conservative estimate, it may not capture the full complexity of adhesive behaviour, especially in this case where adhesive toughness plays a significant role in relation to crossover size and its influence on the estimation of the overall critical SERR $G_c$.

In summary, this research contributes to a better understanding of the impact of parallel defects with finite width and infinite length, on adhesively bonded joints, and offers insights into the critical SERR $G_c$ and crack propagation in such heterogeneous structures. This study emphasizes the importance of considering localized finite defects in predicting the joint behavior of adhesively bonded assemblies.

References


