DOI: 10.2478/v10077-011-0010-z

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TWO TESTS FOR ADHESIVE BONDING LONG TERM CHARACTERIZATION: PRINCIPLES AND APPLICATIONS

ABSTRACT

This article describes recent refinement of the traditional wedge test technique used to characterize durability of the adhesive joints. We propose two types of measuring protocols to monitor precisely and continuously the propagation of an "effective" crack during long term mode I fracture mechanic test. First method is directly derived from the traditional wedge test technique and consist in monitoring the surface strain of adherent with resistive gauges. The second method consist in replacing constant applied displacement by constant force loading and monitoring the beam deflection. Applications of these techniques are presented concerning crack propagation and nucleation monitoring leading to better understanding of the phenomena occurring in the joint subjected to an external load.

Keywords: *adhesion, fracture, wedge test, constant force test, durability.*

INTRODUCTION

The wedge test, originally developed in the 1970's by the Boeing company, remains probably the less expensive method to evaluate, in a reliable way, the long term durability of adhesive bonding. In this experiment, two adherents, considered as cantilever beams, are bonded together prior to insertion of a wedge [1-4]. Constant displacement boundary condition leads to the cleavage in the bondline which may result in crack nucleation and propagation depending on load intensity and mechanical strength of the assembly. Knowing the adherent elastic properties and geometry, and owing to the *beam like* geometry of the sample, simple formulas are used to determine the instantaneous fracture energy G provided the crack position is known [*e.g.* 5]. As a result of this experiment, the crack length increase, as a function of time, is given. More intrinsic bondline properties are given by the relation $G(da/dt)$ *i.e.* instantaneous energy release rate as a function of crack propagation speed. For quantitative analysis of this experiment, precise measurement of crack position is required [6, 7]. First, this geometrical parameter is necessary to calculate the energy released rate, second continuous monitoring of crack propagation is necessary for proper derivation and calculation of speed da/dt which might vary rapidly in the early stage of nucleation and propagation. Up to now, almost no efforts have been done to improve the metrology of this long term crack propagation experiments. The most popular technique still consist in observing the crack tip position with the side view microscope like in Fig. 1, so that most experimental results are limited to qualitative *a(t)* curves [8].

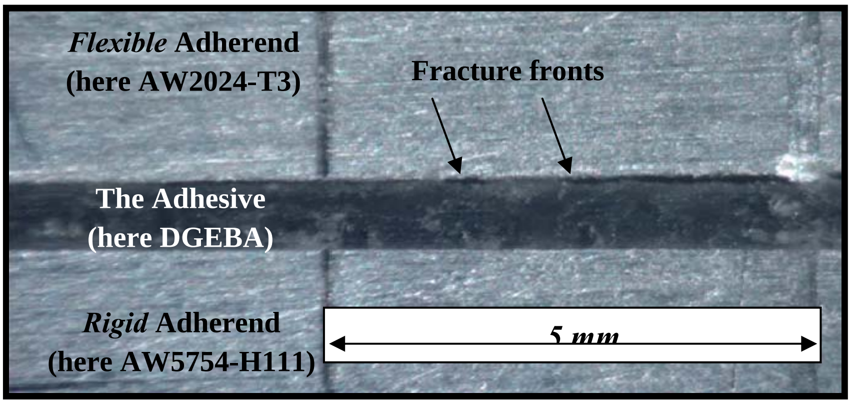

Fig. 1. Crack propagation during wedge test experiment

In an effort to propose a cheap, robust and stand alone test we proposed an experimental arrangement [9] to determine an *effective* crack length position as calculated from the strain measured at the surface of the flexible adherent. Since then this technique has been applied on complex configurations such as variable bonded surface properties [10], or to evaluate the elasticity of the adhesive layer [11]. Apart from systematic error, associated to uncertainty in the initial position of the wedge or material properties, the relative error in variation of the crack length *a* is smaller than 1%. Furthermore this technique permits continuous monitoring of the crack propagation in time. However, constant displacement conditions (NB leading to the quasirelaxation at the test end) might not be the most critical and representative conditions for structural parts. We wished to investigate the long term crack propagation in mode I when applying a constant force. In this configuration the instantaneous, *effective*, crack position is determined from the measurement of the beam deflection with an LVDT (linear variable transducer) sensor using simple formula of beam bending rigidity. This experimental configuration reveals to be more delicate to manipulate and design due to the crack propagation instability when approaching the critical energy released rate. In return, measurements give an additional information concerning the crack nucleation kinetic prior to propagation (*viz*. initial creep). In this configuration, Winkler elastic foundation model is used to take into account the mechanical behaviour of the adhesive layer. Accordingly, part of the article will be devoted to the explanation and application of the elastic layer theory as a part of the analysis of long term crack propagation test. All experiments presented within the paper are performed on the same adhesively bonded system (the same materials used for all substrates). Surprisingly, different propagation kinetic is observed in both tests. These could be, partially, attributed to different, not deliberately (and as we believe less important), introduced variables (different surface conditioning procedures and the adhesive material variability - a commercial adhesive was used to develop these experiments). In contrary, some of the results achieved proves fundamental differences between constant force and constant displacement protocols being at the heart of our studies.

THEORY

Crack position monitoring

All present experiments are done on asymmetric samples consisting of a *flexible* substrate bonded to a second one considered as *rigid*. This configuration is not conventional compared to the classical Double Cantilevered Beam (DCB) geometry but could be found more convenient (it is easier to ensure perfect *dissymmetry* than perfect *symmetry*). Accordingly, using such system makes the whole sample easier to hold and load.

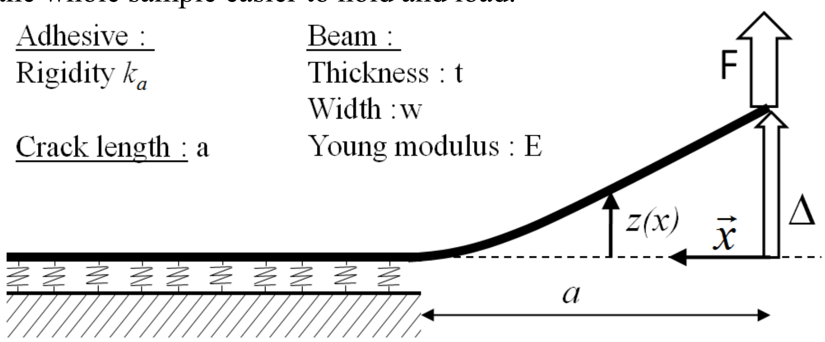

Fig. **2**. Mode I Crack propagation test on asymmetric sample mechanical modelling

Fig. 2 shows physical model of the situation studied. The flexible substrate is loaded in bending, either by applying a define force *F* (for constant force) or define displacement Δ (for constant displacement) at the edge of the unbounded part of the sample. Considering that the flexible beam is *encastré* along the bonded region (which is the standard way of treating the adhesive joints), the sample bending rigidity can be found from the Euler-Bernoulli beam theory:

$$\frac{F}{\Delta} = \frac{Ewt^2}{4a^3} \tag{1}$$

where *E* is the flexible adherent Young modulus, *w* its width and *t* its thickness. *a* corresponds to the length of the unbounded region or equivalently the distance from loading position to the crack front. From Eq. (1), using Griffith theory, energy release rate can derived (corresponding to the mode I loading at the crack front). For the constant applied displacement, fracture driving equation is:

$$G = \frac{3}{8}\frac{Et^2}{a^4}\Delta^2 \tag{2a}$$

In case where the force is controlled, it is more convenient to write:

$$G = 6\frac{a^2}{Ew^2t^3}F^2 \tag{2b}$$

These expressions outline the fast decrease of *G* in wedge test configuration (2a) (constant displacement, *G* proportional to a^{-4}) leading to stable crack propagation, and fast increase of *G* when applying constant force (2b) which makes the crack propagation difficult to control (*G* proportional to a^2).

Instrumented wedge test

Slow rate crack propagation experiments provide an interesting way to characterize quantitatively (and with economical means) the long term behaviour of adhesive joints. Provided the crack propagation rate can be monitored, we obtain quantitative measurements of damage growth (*da/dt*) as a function of loading *G*. To ensure reliable joint performance predictions precise measurement of the crack position is crucial. First, to calculate the crack propagation speed (derivation is required which is very sensitive to *noise*). Second, Eq.(2a) and Eq.(2b), crack length is required to calculate the energy released rate which drives the propagation. In the present configuration, the beam being loaded with a normal force, the bending moment (*M*) vary linearly along the beam (*M=Fx*). As a consequence, the skin longitudinal strain evolution (ε) is also linear ($\varepsilon \sim M$) with maximum being at the crack tip and zero value at the wedge or force position (since $0<x<a$). To monitor this strain, resistive gauges are used in which, according to the Euler-Bernoulli theory, signal should vary with the crack propagation:

$$|\varepsilon(x)| = \frac{3}{2}\frac{t}{a^3}\Delta x \tag{3}$$

Moreover, with several gauges being placed at defined positions x_i with respect to the wedge, a least square minimization can be used to evaluate the slope of strain evolution $\varepsilon(x,t)$ along the bent beam leading to the crack position estimation:

$$a_{app}(t) = \sqrt[3]{\frac{3}{2} t\Delta \frac{\sum_i x_i^2}{\sum_i \varepsilon(x_i, t) x_i}} \tag{4}$$

Assuming perfect built-in condition at a_{app} this experiment is a validation of the Euler-Bernoulli theory, which is demonstrated to be satisfying in our application [**9, 10**]. An important feature of this method is that the crack position becomes a phenomenological/effective parameter. As can be seen in **Fig**. **1**, in many adhesives joints the crack propagation front, at the adhesive layer scale, corresponds to a continuous voids nucleation, growth and coalescence processes. Other systems leads to crazing mechanisms or multicrack development making crack front to be found almost impossible. The *a* estimated with simple beam theory (a_{app}) is an *effective* crack position which growth continuously and enables to measure damage development, whatever is the mechanism! (although with different physical meaning).

Constant force test

Main difficulty in the wedge test experiment arise from the necessity to evaluate the applied force so that the sample itself must be changed into a load cell. To overcome this difficulty, simple modification in the experimental protocol consist in replacing the constant displacement condition by constant force. This can be easily achieved by suspending a mass at the tip of the flexible beam [**12**]. With the force being known, the crack propagation can be monitored by measuring, with a displacement sensor, the evolution of beam deflection at the loaded point (to maximize signal-to-noise ratio). From Eq. (1) the instantaneous crack position is determined using relation:

$$a_{app}(t) = \sqrt[3]{\frac{1}{4} Ewt^2 \frac{\Delta(t)}{F}} \tag{5}$$

Surprisingly, the sample design and the choice of experimental condition are much more delicate, when compared to the constant displacement set-up, since the crack propagation may become unstable with G being proportional to a^2 and F^2 in Eq.(2b). Initial crack length and applied force are very critical in this experiment. If load or crack length are too high, the slow rate crack propagation domain might be missed, and unstable propagation occur immediately. If these parameters are too small, propagation might never occur. Besides, when crack length increase, maximum stress in the flexible beam also increase and may overcomes yield stress of the material. To estimate maximum acceptable crack length, minimum beam thickness has to be calculated so that global behaviour remains elastic and leaving Eq.(5) valid. Last criteria in the design of the experiment concerns the range of the displacement sensor. Range must be adjusted to optimize the measurement precision up to the end of the test (when maximum deflection Δ_{max} is expected). With these considerations we can optimize the experimental conditions assuming flexible beam geometry and the joint fracture energy [G_{min} G_{max}] interval. Maximum crack length ≈ sample length is given by relation:

$$a_{\max} = \left(\frac{3E}{8G_{\max}} t^3 \Delta_{\max} \right)^{1/4} \tag{6}$$

The applied force is equal to:

$$F = \frac{E\Delta_{\max}}{4a_{\max}^3} t^3 \tag{7}$$

The initial crack length:

$$a_{\min} = a_{\max} \sqrt{\frac{G_{\min}}{G_{\max}}} \tag{8}$$

The maximum stress at the end of the test:

$$\sigma_{\max} = \frac{6Fa_{\max}}{wt^2} \tag{9}$$

For instance, in case when σ_{max} overcomes the material yield stress the range [G_{min} G_{max}] has to be modified or if possible the adherent thickness must be increased.

EXPERIMENTAL EXAMPLES AND ANALYSIS

In all tests samples made from 2024 aluminium plates were bonded together with a commercial epoxy adhesive Aralidte Crystal (Bostik, France) consisting of the DiGlycidyl Ether of Bisphenol A (DGEBA) resin, cured with an amine crosslinking agent at ambient temperature. Flexible adherent was of thickness t=1.6 mm with rigid adherent being 5 mm thick. Both plate were of width w=25 mm (as recommended in wedge test experiments). Adhesive thickness, as measured with optical microscope, is 350 μm. For the wedge test two systems were considered, the difference being surface treatment a priori to bonding. In the first case simple abrasion with 400 grade emery paper was performed. In the second abrasion was followed by Phosphoric Acid Anodization (PAA) (10V DC, 20 min in ambient temperature). During the test flexible adherent strain was measured with 3 resistive gauges to monitor the crack propagation. Objective of the present development is not to focus on material aspects but on the new possibilities offered by this instrumented technique with the first one being continuous monitoring of the crack propagation. In Fig. 4 instrumented wedge test results are shown.

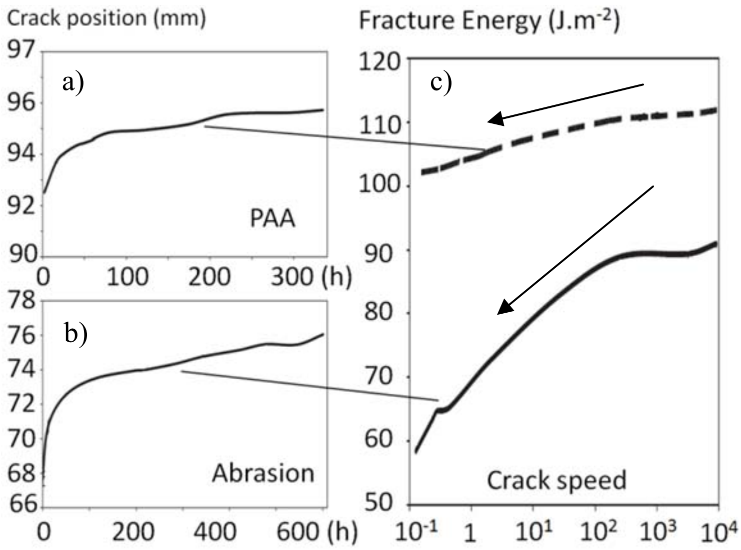

Fig. 4. Instrumented wedge test characterization of aluminium/DGEBA bonded joint. Comparison between electrochemical (PAA) surface treatment and manual abrasion. a) Crack increment for electrochemical treatment, b) the same as a) but for abraded surface, c) fracture energy vs. crack speed

It must be emphasized that the sensitivity, to the relative variation of a, was proved [**9**] to be high. These two measurements clearly distinguish the behaviour of both systems, but over all enables to observe very weak fluctuations in the $G(da/dt)$ that may be attributed to diverse phenomena (local heterogeneities).

Almost similar to the previous bonded system was used in the constant force arrangement. In this test, flexible adherent width was w=5 mm. The second difference was surface treatment with surfaces being now sandblasted and anodized with a sulfuric acid-based treatment. All important evolutions are presented in **Fig**. **5** (beam deflection Δ, rigidity K, crack length, a and fracture energy G). Again, the important aspect of this method is the possibility to monitor continuously the damage development in the bondline associated to the growth of an effective crack length.

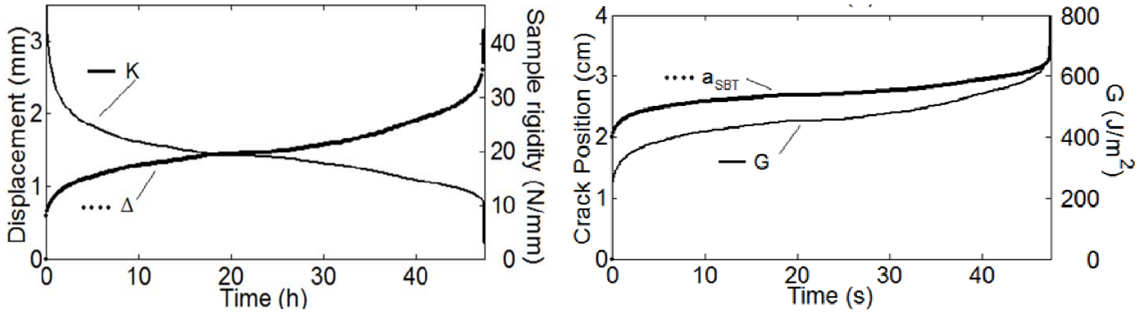

Fig. 5. Crack growth monitoring in Constant Force Test, in epoxy bonded aluminium joint

In Fig. 6 estimated fracture energy (G) is given as a function of the crack speed. This measurement points out fundamental difference between constant displacement and constant force measurements. Contrary to the wedge test where G is only decreasing with da/dt (Fig. 4c) in constant force system initial increase of the *energy* with decreasing speed is observed. We found this to be associated with the crack nucleation prior to the crack propagation period (not visible with wedge test). It must be appreciated, that in this case, using Griffith criteria for fracture, is not correct physically. Elastic energy is not released in form of crack increment but in form of the adhesive viscous flow.

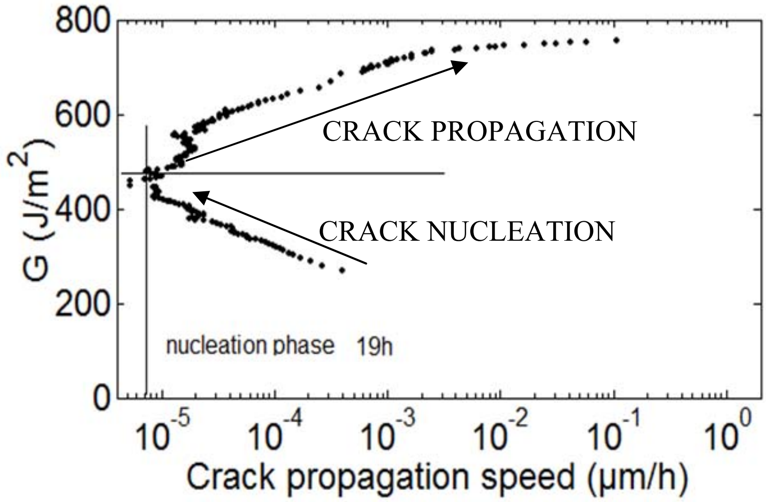

Fig. 6. Crack growth kinetic as measured with CFT

Accordingly, to investigate this phenomenon, the simple beam analysis is not sufficient since the adhesive layer is supposed to be perfectly rigid.

Winkler elastic foundation

Simple way to take into account bondline layer is the Winkler elastic foundation theory. In this model, the foundation reaction f is proportional to the local beam deflection z with relation:

$$f = -kz \tag{10}$$

where E_a, ν_a and t_a are respectively the adhesive Young modulus, Poisson coefficient and adhesive layer thickness. k quantifies the adhesive layer rigidity. The flexible beam equilibrium equation above the bonded area becomes now:

$$\frac{d^4 z}{dx^4}+\frac{k}{EI}z=0 \tag{11}$$

From the resolution of this differential equation, and global mechanical problem, we find the flexural rigidity of the beam on elastic foundation:

$$\frac{F}{\Delta}=\frac{Ewt^3}{4a^3}\frac{2(a\lambda)^3}{(3+6\lambda a+6\lambda^2 a^2+2\lambda^3 a^3)} \tag{12}$$

where:

$$\lambda a = a\frac{\sqrt{2}}{2}\left(\frac{k}{EI}\right)^{1/4} \tag{13}$$

is a nondimensional number indicating the ratio between the crack length and the stressed region in the adhesive. From Eq.(12) the expression of the energy released rate in constant displacement configuration is found in form:

$$G=\frac{3}{8}\frac{Et^3}{a^4}\Delta\frac{4(\lambda a)(1+\lambda a^4)^2}{(3+6\lambda a+6\lambda^2 a^2+2\lambda^3 a^3)^2} \tag{14}$$

or, equivalently, in constant force configuration:

$$G=6\frac{a^2}{Ew^2 t^3}F^2\left(1+\frac{1}{\lambda a}\right)^2 \tag{15}$$

The postfactor in Eq.(14) and Eq.(15) can be considered as correction on G to take into account the elasticity of the adhesive layer which can be calculated providing the effective adhesive layer rigidity k_a is measured or calculated. If not, Eq.(14) and Eq.(15) show that correction on G is not necessary if parameter λa is larger than ≈ 10. As a consequence, simple beam theory is convenient when the crack length is large compared to $1/\lambda$. To increase coefficient λa crack length can be increased and/or flexible beam thinned. For simplicity it is convenient to use simple beam theory formulas to evaluate the crack position in instrumented wedge test experiment so as in constant force test experiment. The ratio between estimated effective position and geometric *real* crack length in this two experimental configuration is equal to:

$$\frac{a_{app}}{a}=\sqrt[3]{\frac{2(\lambda a)^3+6(\lambda a)^2+6\lambda a+3}{2(\lambda a)^3}} \tag{16}$$

showing that with both techniques crack length is generally overestimated. In usual, properly chosen experimental conditions, error is less than few percent which can be satisfying since very simple experimental configuration and analysis methods are used.

Analysis of IWT with the Winkler model

To prove and validate Winkler-based model the beam deflection in the process zone (right behind the crack) was measured using an Altisurf®500 profilometer (Altimetr, France). Profilometry results, corresponding to the joint with the PAA surface treatment is shown in Fig. 7.

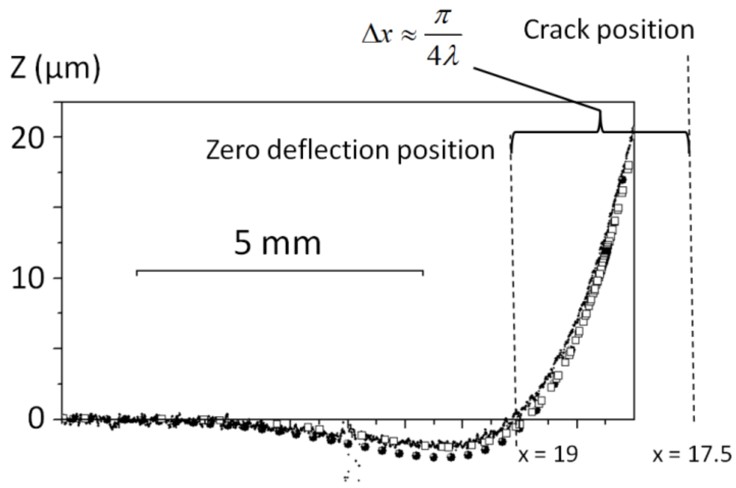

Fig. **7**. Experimental evidence of process zone in front of crack. Interferometric measurement of beam deflection above adhesively bonded region

Clear evolution of the deflection can be found in front of the crack in the bonded zone. Complete resolution of Eq.(11) enables to evaluate this deflection so that we find:

$$z(x)\Big|_a^{+\infty} = e^{\lambda(a-x)}\left[A_1 \cos\lambda(a-x) + B_1 \sin\lambda(a-x)\right] \tag{17}$$

with:

$$A_1 = \Delta \frac{3(1+\lambda a)}{(3+6\lambda a+6\lambda^2 a^2+2\lambda^3 a^3)} \tag{18a}$$

$$B_1 = \Delta \frac{3\lambda a}{(3+6\lambda a+6\lambda^2 a^2+2\lambda^3 a^3)} \tag{18b}$$

The result fits well with the Winkler foundation in which shear elasticity can also be taken into account [13]. Accordingly, this technique allow us to determinate the process zone size ($\lambda^{-1}=4\Delta x/\pi$ with Δx being the distance between maximum and 0 strain as shown in Fig. 7) to be of about 3 mm (in ambient conditions, stationary crack). By slightly modification of our Instrumented Wedge Test the process zone can also be monitored during the crack propagation [12] providing that the strain gauges are bonded in front of the crack front and are passed by the crack during the test. Example of such situation is shown in Fig. 8 in which to provoke crack propagation (from the arrested position in ambient conditions), temperature (from ambient to 40°C) was increased so that one of the strain gauges ($\varepsilon_s(x_{10})$) was passed.

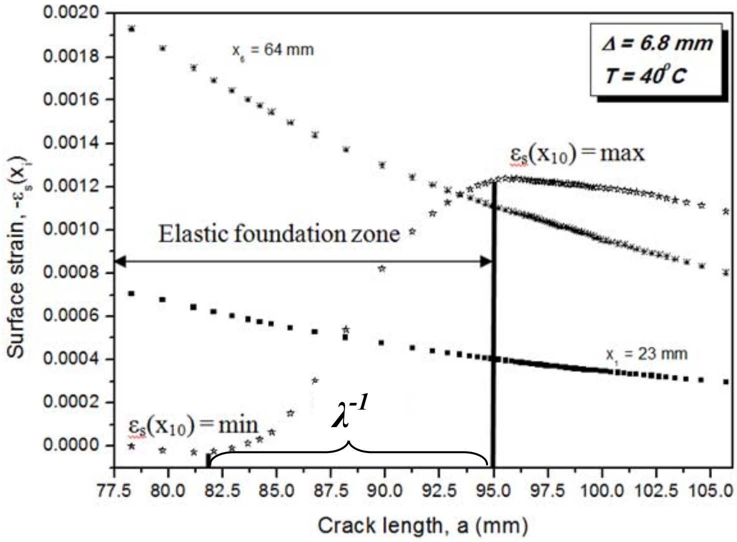

Fig. 8. Evaluation of the process zone size with instrumented wedge test experiment

These strain measurements reveals larger process zone of *ca*. 12.5 mm when compared to the one observed with interferometric measurements. This process zone increase shows impact of the temperature on the adhesive state (increase of viscosity, decrease of rigidity). More importantly this result proves that the adhesive state can be followed by the test introduced potentially leading to better understanding of fracture as well as estimations of crack position and *G* in hostile environments.

Evidence of the crack nucleation in CFT

The elastic foundation model can be used for analysis of the nucleation stage during constant force experiment for which no physical decrease of da_{SBT}/dt with time was observed. This behaviour can be attributed to the viscoelastic strain increase which leads to crack nucleation prior to propagation [14]. Considering viscoelastic behaviour the adhesive rigidity now corresponds to the instantaneous $f(t)/z(t)$ ratio in Eq.(10). As a consequence, the parameter $\lambda(t)$ becomes also function of time, so that the initial apparent variation of crack position should be attributed to viscoelastic creep mechanism rather than crack propagation process. Assuming in Eq.(16) that crack length $a=a_0$ remains constant during nucleation, the observed evolution of a_{SBT} can be used to evaluate the evolution with time of parameter $\lambda(t)$ which describe the viscoelastic damage in the process zone. In Fig. 9 results of the $\lambda(t)^{-1}$ analysis as well as increment of apparent crack position (Δa_{SBT}) are shown in logarithmic scales.

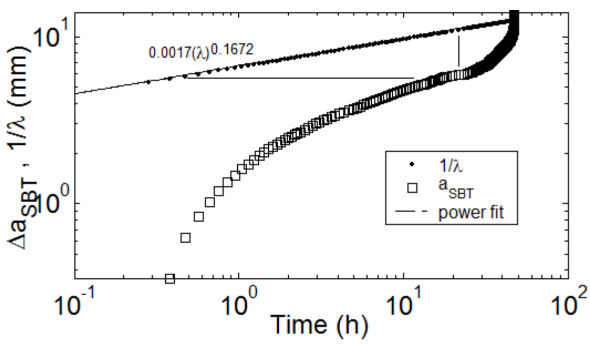

Fig. 9. Crack growth nucleation in the constant force test

The two regimes are clearly distinct when looking at a_{SBT} evolution since the transition between nucleation and propagation stage is marked with an inflexion point. Surprisingly, evolution of $\lambda(t)$ is linear on this graph suggesting clearly power law relation. Also three important parameters can be determined from such analysis. First, the initial λ_0^{-1} value ≈ 4.5 mm (which stays in good agreement when compared to the one found with IWT) and corresponding to stationary crack and t=0. Second, the critical λ_c^{-1} ≈ 10 mm value (prior to crack propagation which is very close to the one observed in wedge test configuration with strain gauges measurement but at higher temperature). Finally, power law parameters describing the kinetic of crack nucleation which in this experiment fits well with a power law is:

$$\lambda^{-1} = 601t^{-0.1672} \tag{19}$$

It must be noted, that all these phenomenological parameters *a priori* depends on the value of constant applied load.

CONCLUSIONS

Within this contribution we have proposed two promising methods and their potential to investigate the crack nucleation and crack propagation in the structural adhesive joints. This low cost, standalone techniques offers attractive possibilities to characterize the long term behaviour, and potentially durability of the adhesively bonded joints. Both methods are based on classical mode I crack propagation except that asymmetric bonded joints are preferred to the classical double cantilevered beam (DSB) samples. Two experimental configurations associated to two distinct loading conditions are proposed. The first consist in using strain gages, in classical wedge test experiment, to cope with the difficulty in measuring of the applied force. In this experiment the flexible adherent is used as a specific load cell reacting on any change in the adhesive state, or crack propagation. In the second configuration, a constant force is applied with suspended mass so that beam deflection is now monitored to detect, *a priori*, crack propagation. Modelling adhesive layer with the elastic foundation Winkler theory enables us to introduce an additional parameter, $1/\lambda$, characterizing the size of the process zone in front of the crack and directly related to the adhesive properties and state. Since the apparent rigidity of the adhesive joint can be also affected by viscoelastic creep/relaxation mechanism in the adhesive process zone due to introduction of $1/\lambda$ virtually we get access to (any) damage mechanisms involved being important advance in testing of the adhesive joints.

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