The Adhesive Zone Mix Disbond Arrest Feature – Results (EU-FP7 Project BOPACS)

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ABSTRACT

Nowadays bonded joints are state of the art in the assembly of composite aircraft structures. Due to current civil aerospace regulations, the implementation of secondary bonded joints is limited. However, current regulations and means of compliance provided by the authorities offer four possibilities to attain certification of safety critical structures: a fail-safe joint design, full proof testing, non-destructive testing of the joint strength, and a secured maximum disbond by design features assuring a limit load capacity of the remaining joint. The last approach has been investigated thoroughly in the European 7th Framework research project BoPAcS (Boltless Assembly of Primary Aircraft Structures). The presented paper summarizes the activities and results of the German Aerospace Center (DLR) within the BoPAcS project. In particular, this paper focuses on the so called Adhesive Zone Mix (also known as "Hybrid Bondline") Disbond Arrest Feature (DAF) developed by DLR. The feature efficiently combines an adhesively bonded and a thermoplastic welded joint. The thermoplastic weld stops growing cracks due to its high toughness while the conventional adhesively bonded joint carries the major operational loads. The paper outlines the smart manufacturing process enabling an easy to use and low cost Disbond Stopping Feature. The mechanical effectiveness is validated on coupon level with fatigue testing of cracked lap shear (CLS) samples. Experimental and analysis results are summarized. Conclusions from tests and the development is given. The concept is protected by patent (DE 10 2013 107 849).

1.0 INTRODUCTION

1.1 Motivation

Adhesively bonded joints offer major advantages to composites if compared to bolted joints, since they lead to weight reduction, offer a more uniform load distribution, are capable of joining thin-walled parts, and minimize material weakening. Development of adhesive bonds being capable for certification of primary aircraft structures is of high interest to the industry. The implementation of bonded joints in primary aircraft structures is still limited due to certification requirements. Normally, certification compliance is managed by so called "chicken rivets" if used in primary parts [1].

As a consequence of negative experience in the past, authorities, namely the Federal Aviation Administration (FAA, USA) and the European Aviation Safety Agency (EASA, Europe), dictate two major prerequisites to be met in order to achieve certification. [2,3]. The manufacturing process must be specified, controlled and monitored and has to be carried out in a pre-defined manufacturing process window regarding influencing parameters. Additionally to the quality management, one of the following methods has to be established to demonstrate certification compliance:

1. Disbonds larger than a pre-defined maximum must be prevented by design features. The allowed disbond maximum must be determined by analysis, test, or both.
2. Proof testing has to be executed for every production article to ensure that the joint can withstand the desired design loads.
3. The load-bearing capability of each joint must be determined by repeatable and reliable non-destructive inspection (NDI) methods.

Proof testing of each production specimen is not feasible in serial production of composite structures since testing is very cost-intensive. Typical manufacturing defects are detectable by today's methods like ultrasonic scanning or log-in thermography. An NDI method that is capable of measuring the adhesion is up not in sight. Finally, a promising approach is to use disbond stopping design features. Those must be developed and incorporated in each bond to prevent a possible disbond reaching a critical extent [4].

The European research project BOPACS was formed to identify and investigate solutions for new disbond arrest features in order to overcome current certification limitations.

1.2 The European FP-7 Project BOPACS

The European research project "BoPAcS – Boltless Assembly of Primary Aircraft Structures" [5] was funded within the Framework FP7 by the European Commission. It started in September 2012 and ended in October 2016. Thirteen European partners, led by Airbus Operations (Germany), worked together to identify and investigate solutions for new disbond arrest features. BOPACS's main objectives are:

1. To reduce weight and costs of primary aerospace composite structures by developing bolt free adhesive bonded joining that comply with the airworthiness requirements

2. To design and assess crack stopping design features limiting the maximum disbond size in adhesively bonded joints as a mean of comply (MOC) to airworthiness authorities requirements

Five different categories were identified for disbond arrest features: "Through thickness reinforcements", "Surface and geometry modification", "Surface interfacing features", "Adhesive bondline architecture", and "Supporting adhesive modification".

DLRs work focussed on two different disbond arrest features. The first concept is a combined bonding and bolting with small diameter pins [6]. The second concept is the *Adhesive Zone Mix Disbond Arrest Feature* (further called *Hybrid Bondline)* [7] consisting of a conventional adhesive and welded thermoplastic. The present paper summarizes the project results of the second concept.

2.0 THE HYBRID BONDLINE

2.1 Concept

Typical epoxy adhesives used in aerospace structures are toughness-modified to reduce brittle behaviour. But adhesive toughening by integration of e.g. rubber particles causes a reduction in stiffness and strength. Toughening is a compromise of damage tolerance and increase in stiffness or strength, respectively. The hybrid bondline concept (Adhesive Zone Mix within the project) installs multiple adhesive regions by combining different adhesives with altering properties. A strict separation of both functions: toughness for crack-stopping, and shear strength and stiffness for load transfer is of uttermost importance for the certification compliance. This is achieved by separating the bondline into several areas as shown in Figure

21-1. The disbond arrest feature is applied in a discrete manner at distinct positions within the bondline [8].

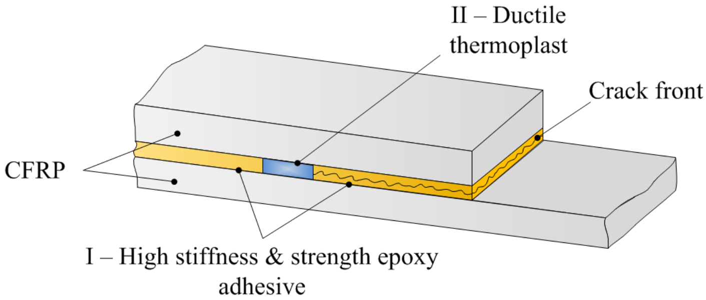

Figure 2.1-1: Hybrid Bondline: Principle alignment of both adhesives within the bondline for a single overlap configuration

On the one hand, a conventional high-strength structural adhesive is used in phase I (Figure 2.1-1). Due to its high stiffness compared to phase II, major loads are carried by phase I. On the other hand, phase II shall be realized by the implementation of a pure thermoplastic material showing superior ductility. Accordingly, this physical barrier acts as a disbond-stopping feature (DSF) within the bondline through a sharp change of materials as the epoxy and the thermoplastic are not soluble. An interface is created between both materials. As adherends and adhesive remain unaffected, weakening of the joint is not expected. With this concept, a new crack initiation would be needed for further crack growth, which is unlikely in or behind the rather ductile thermoplastic material. Consequently, fatigue resistance after crack stop should be significantly increased. A hybrid bondline is formed by combining two different joining techniques: adhesive bonding and thermoplastic welding. For the weld, the so-called thermoset composite welding (TCW) shall be used. Thus, the joint does not solely rely on adhesive bonding. A weak bond within the welded interface of the DSF is less likely to occur since welding is not prone to surface contamination as adhesive bonding, as it does not rely on adhesion. The nature of the co-cured interface is investigated in section 3.1

2.2 Materials

For composite adherends an off-the-shelf prepreg system supplied by Hexcel is used. It consists of 8552 matrix resin and IM7 carbon fibers with a ply thickness of 0.125 mm [9]. The material is chosen for all experimental studies due to its widespread implementation in aeronautical applications. An epoxy film adhesive material supplied by Henkel, named Loctite EA9695 0.05 PSF K (here after referred to as EA9695), is used as a common high-strength adhesive for aerospace applications [10]. Film adhesives are widely used for composite joints, for instance for co-bonded joints of stiffeners in the Airbus A350 or composite repairs. PVDF Kynar 740 supplied by Arkema is used as it shows high mechanical strength and stiffness, a comparatively low melting temperature of 167°C, and high ductility with an elongation at break between 50% and 200% [11]. Material of 0.125 mm in thickness is used since this thickness is comparable to conventional epoxy film adhesives.

2.3 Processes

The joint manufacturing can be divided into four steps, as shown in Figure 2.3-1. First, the thermoplastic strips are put into place before curing of the adherend parts (Step I). The prepreg curing cycle is conducted as specified by the material data sheet. Since the thermoplastic melts already below the curing temperature of the matrix resin, liquefaction of the thermoplastic during the process occurs. As both systems (8552 resin and PVDF) are simultaneously liquid during the curing process as defined by the data sheet [9], a strong bond is expected between the thermoplastic and the composite's matrix system. It is believed that a semi-interpenetrating network between both materials is established. Since this interface is crucial for the overall strength of the thermoplastic barrier, the adhesion mechanism is studied in more detail in section 3. The thermoplastic strips are placed on both adherend surfaces. Thus, the strips face each other when both parts are joined. In step III the actual bonding is conducted by placing the epoxy film adhesive on one adherend with the area of thermoplastic strips left open. Two additional strips of the thermoplastic with an overall thickness of 0.25mm are placed in the area left open. In the final step IV (Figure 2.3-1), the adhesive is cured and the bond is thus established. In parallel, thermoplastic welding is obtained to achieve a mechanical resilient PVDF area. In doing so, all four thermoplastic strips are welded through coalescence of the material. The high temperatures at the interface lead to healing of the material and thereby to a homogenous polymer network

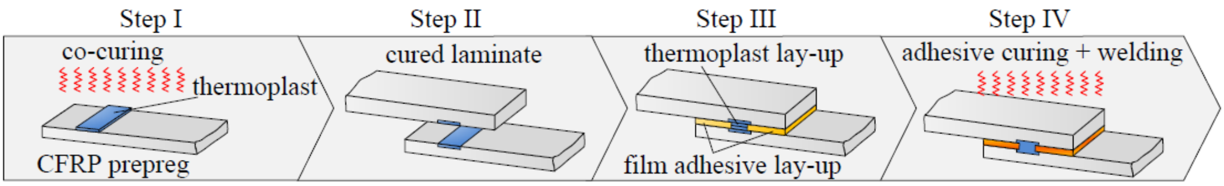

Figure 2.3-1: Manufacturing steps for hybrid bonded and welded joint (Hybrid Bondline)

3.0 INTERFACE CHARACTERIZATION

A crucial part of TCW joints is the adhesion mechanism of the co-cured thermoplastic layer on top of the thermoset matrix resin, i.e. the first laminate ply, as the joint's performance is highly dependent on the interface region of the two dissimilar materials. The transition between both materials is addressed in section 3.1.

3.1 Material transition between PVDF and Matrix Resin

The occurrence of interdiffusion of molecules is typically in a range between 1nm and 100 nm and depends on molecular weight, structure, temperature, and the thermodynamic miscibility of the polymers [12]. High resolution light microscopy images indicate a rather narrow transition and no distinct interdiffusion zone. Therefore, a scanning electron microscope (SEM) is used to examine the transition with higher magnifications. A detailed analysis of the transitions zone (Figure 3.1-1 (left)) does not provide any information regarding a possible interdiffusion zone. Even improved SEM pictures produced by focused ion beam preparation show no interdiffusion zone (Figure 3.1-1). As another analysis method, energy-dispersive X-ray analysis (EDX) is applied to study the material transition. Since PVDF is a fluorine-containing thermoplastic, the fluorine content can be used as a marker to determine whether interdiffusion takes place. A change of fluorine content from about 5% to about 35% is measured by EDX analysis within 1μm by examining the transition zone between 8552 matrix and PVDF as shown in Figure 3.1-1 (right)

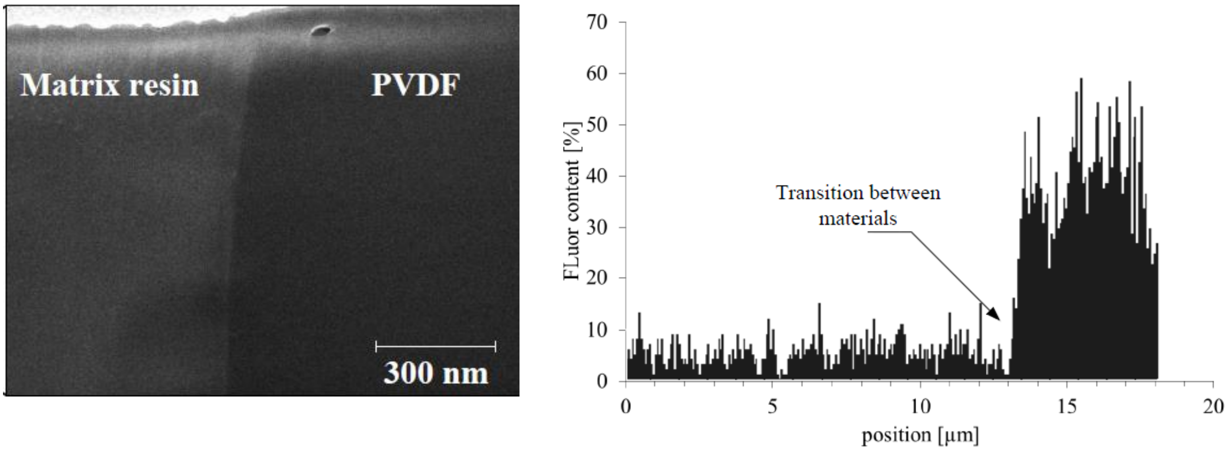

Figure 3.1-1: FIB cut area (left) and transition zone between PVDF and epoxy matrix resin (left), EDX measurements of fluorine content [%] at the transition of both materials;

It can be concluded that evidence of these adhesion mechanisms remains challenging. Nevertheless, evaluation may be done indirectly by mechanical tests that are suitable to assess of adhesion quality. In all manufacturing an experimental studies it can be stated that the PVDF-epoxy interface was never the weak link

4.0 STATIC STRENGTH

4.1 Double Cantilever Beam Specimen with Hybrid Bondline

Double Cantilever Beam (DCB) specimens are used to evaluate the crack stopping capability of the hybrid bondline approach under pure mode-I (peel), quasi-static loading condition. In contrast to other typical quasi-static tests, this peel load configuration allows continuous crack growth without the need for cyclic loading, which brings a great reduction in test effort. Additionally, a test in pure mode I is considered as a critical benchmark for the DSF, as adhesive joints are generally prone to this type of loading. Hence, positive crack retardation effects are highly desirable. The PVDF area is inserted 65 mm from the loading point of the piano hinges as shown in Figure 4.1-1.

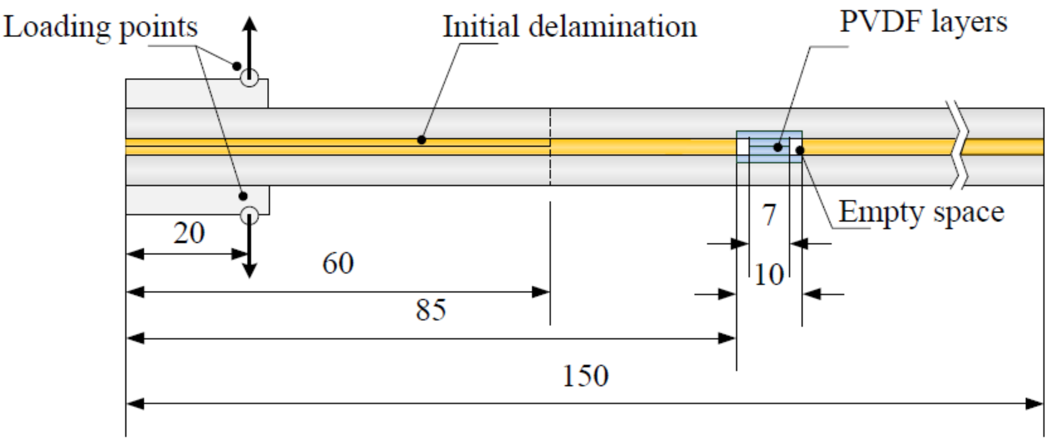

Figure 4.1-1: DCB specimen with implemented crack stopper (dimensions in mm)

4.2 Manufacturing

Curing, bonding and welding of the 7 25 mm wide specimen is done in an autoclave and described in [4]. The integrated strips of PVDF that are co-cured with the laminate are 10 mm in width. The 2 additional strips that are placed in between both adherends (i.e. the actual bondline) are 7 mm in width. The empty space beneath the strips allows some material flow in order to reduce the initial thickness of 0.25 mm to approximately 0.15 mm, thus in the range of the epoxy thickness after curing. Assuming that the density of PVDF remains unaffected by the process, a reduction in thickness to 0.175 mm is expected to occur due to material flow into the empty space.

4.3 Testing

Testing is done with a testing speed of 2 mm per minute as specified by the respective test standard ISO 25217 [13]. Test results are compared with reference samples that are bonded without DSF. Since stick-slip behaviour occurs for some specimens, only the simple beam theory method is used for the determination of fracture energy values. Although the tests are carried out in two consecutive steps, the pre-cracking stage in which crack movement occurs from the inserted release film into the adhesive is not described here.

4.4 Results

An influence of the PVDF area is clearly visible by load-displacement curve of the reference and the applied load, as depicted in Figure 4.4-1 (left). Both the reference and the DSF samples look somewhat similar for 1 mm to 12 mm of cross head displacement. Hence, no negative influence of the crack stopper is observed.

Subsequently, a load drop occurs for the PVDF samples, whereas the reference samples are characterized by a steady decrease in load with increasing displacement (Figure 4.4-1 left). Micro sections do not reveal any voids or signs of false processing of the epoxy. DSC measurements prove that the PVDF and the adhesive are not fluid at the same time. The PVDF is incompressible during the whole proves and does not change its thickness during the bonding process. The pressure distribution during bonding may be different in the vicinity of cracks topper resulting in a lower performance of the adhesive. For displacements larger than 12 mm, a steady load increase is observed for DSF-containing specimens, until a sudden load drop occurs at the point of failure for rather high displacement values of 25 mm to 30 mm.

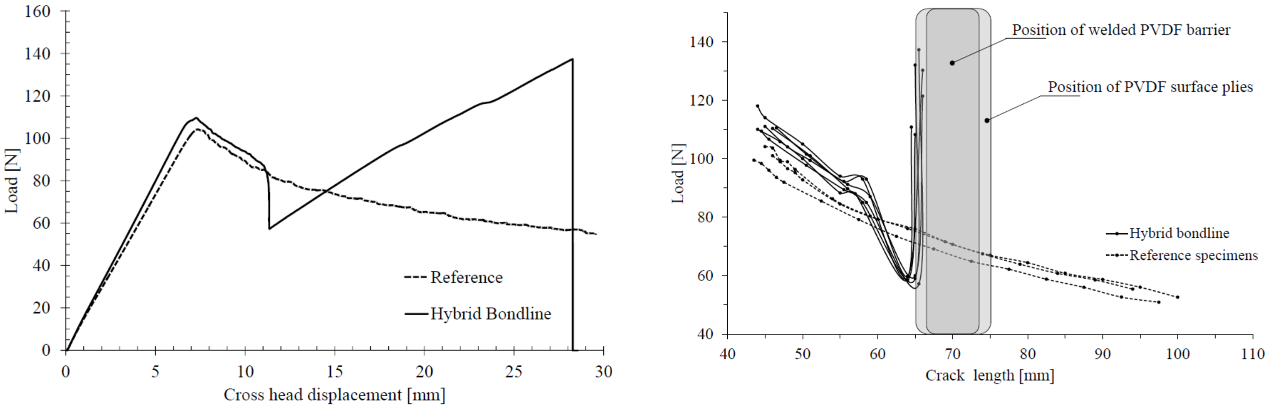

Figure 4.4-1: Load-displacement curve of reference vs. hybrid bondline DCB specimen (left), load-crack length curve of reference vs. hybrid bondline DCB specimen (left)

The influence of the PVDF phase becomes more obvious by plotting the applied load versus measured crack length ((Figure 4.4-1 right)). Up to a crack length of 55 mm no influence is evident and the crack growth behaviour is comparable to the reference samples. When the PVDF barrier is reached (65 mm of crack length), very large loads are needed to force further crack growth. An average value of 124.8 N (69% above the reference value for the same crack length) is measured before spontaneous overall unstable failure occurs. By calculating the Strain Energy Release Rate (SERR) for this initiation point of further crack growth, a value of 2075.3 J/m^2 is obtained which is close to the pristine material fracture toughness.

The DCB results demonstrate the crack-stopping capability of the concept under mode I loading conditions. The crack growth is significantly retarded by the presence of the welded PVDF barrier and is shifted to noticeable higher loads. However, a decrease in the resistance to crack growth is observed for approximately 5mm ahead of the PVDF zone. This is believed to be caused by a slight increase in the adhesive thickness in the vicinity of the PVDF area, which typically accompanies a decrease in fracture toughness [14].

5.0 FATIGUE RESULTS

5.1 Fatigue Test Campaign

The crack lap shear (CLS) specimen is chosen, as shown in Figure 5.1-1. The configuration is especially representative for long, low-loaded overlap joints such as doubler to skin configurations. In contrast to DCB tests, tensile deformations could be set to a realistic magnitude with respect to in service loads. In fact, there are no standardized procedures on the experimental fatigue of bonded composite joints available in general [15]. Nevertheless, numerous experimental studies have been carried out in the past. Hence, the dimensions are chosen based on available literature [16,17]. The overlap length of 200 mm includes 50 mm of clamping area. The mixed mode ratio (MMR) of G_I to G_{II} for an equal thickness of strap and lap amounts to 0.27 according to beam theory [17]. The position of the disbond arrest feature is set to a distance of 25 mm from the overlap edge.

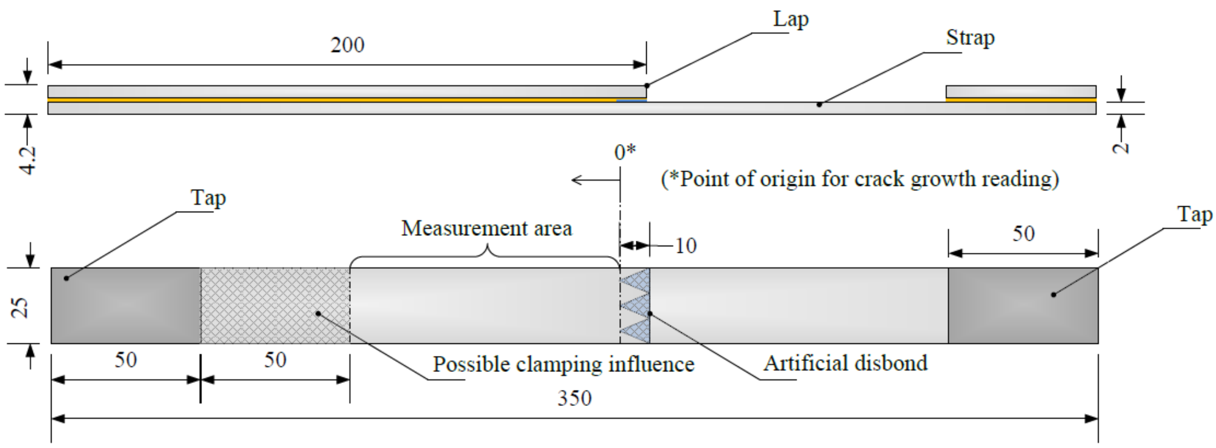

Figure 5.1-1: Geometry of CLS specimen

Plate manufacturing and bonding for the test samples are done in an autoclave cycle and similar to the DCB samples. The fatigue tests are carried out load-driven in a sinusoidal tensile-tensile loading regime. Both tap areas are fully clamped. The R value is set to R = 0.1, as commonly done for fatigue tests of bonded joints, e.g. in [18,16,4]. The fatigue tests are carried out at room temperature conditions using a servo-hydraulic testing machine. Based on the DMA results a test frequency of 8 Hz is chosen as a trade-off between testing time and strain-rate hardening effects. Two microscopes, one for each side, are used to measure the crack length edgewise. The crack measurement is performed every 28 800 load cycles. A maximum of 10^6 load cycles are tested. Two different load levels are tested, 3000 µm/m and 5000 µm/m. The first load equals a typical static limit load criterion; the second is comparable to ultimate load. Note that common fatigue loadings are typically much lower than limit load. Detailed information on the reference specimen is published in [19]. Three specimens are tested for each configuration.

5.2 Fatigue Results

A crack length (a) versus load cycles (N) plot for the load level equal to 3000 µm/m strap strain is given in comparison to reference samples in Figure 5.2-1. For the first 10mm of crack growth, no influence of the DSF becomes obvious. Between a crack length of 10 mm and 15 mm, the maximum averaged crack growth rate for DSF-containing samples amounts to 0.19 µm/cycle and is thus above reference. However, as soon as the crack-stopping area is reached, the crack growth completely stops. Accordingly, no crack growth rates can any longer be given.

The maximum crack length amounts to 15 mm for the 3 samples and is thus ahead of the welded area and just at the edge to the PVDF surface ply. It is therefore assumed that the local surface toughening already positively affects the crack growth behaviour. In fact, numerical studies of Ashcroft et al. [18] indicate that stresses within the adhesive are highly uneven in the thickness direction. Maximal stresses occur at the interface between the strap and the adhesive.

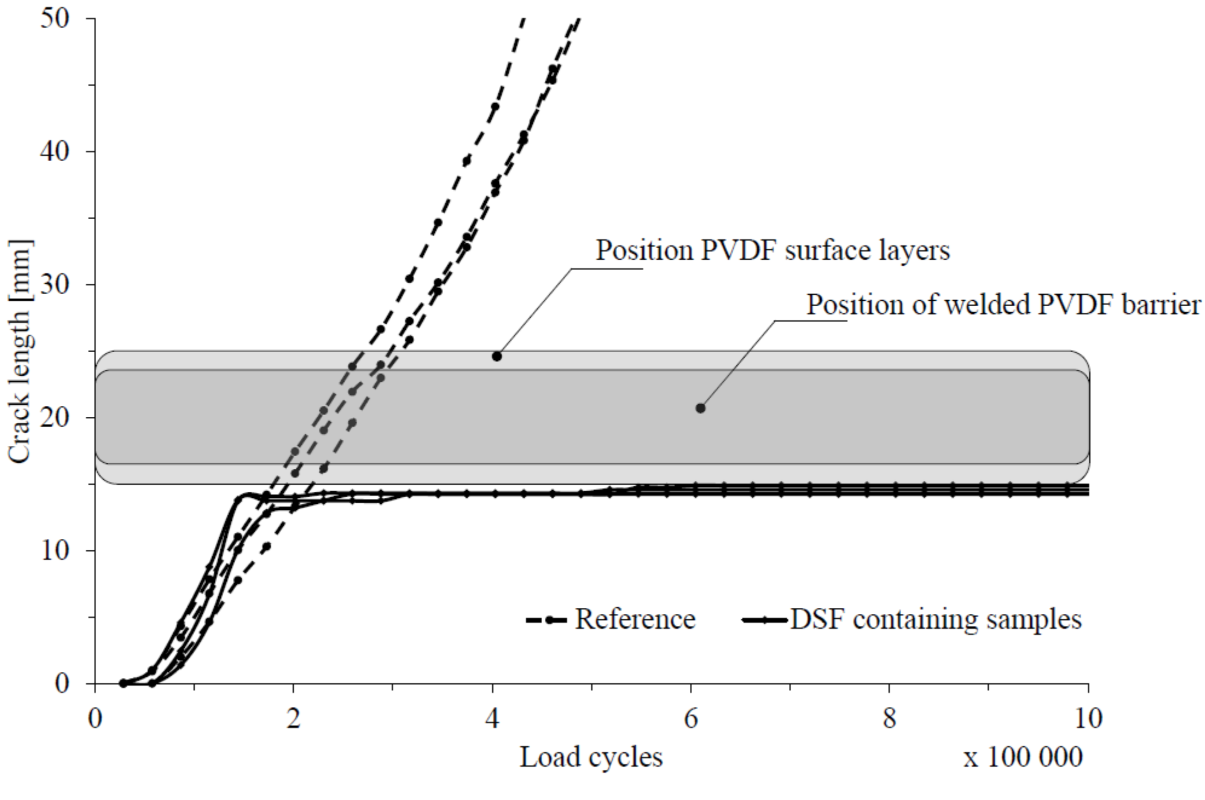

Figure 5.2-1: Maximum load 9.28kN (3000µm/m): Crack length versus load cycles for reference samples and DSF-containing samples

The results for the highest load level (maximum load of 15.6 kN) are given in Figure 5.2-2. As for the lower load levels, no unstable crack growth is observed when the crack-stopping area is reached. For 1 sample out of the 3 crack growth is comparable to the results given in the previous paragraph: the crack growth securely stops at the edge of the welded PVDF area and no delamination is observed. The other 2 samples show a different behaviour, as delamination is observed beneath the welded PVDF strips within the strap. This interlaminar crack is almost connected to the crack front within the adhesive and is thus taken into account for the given results.

The delamination does not start at the interface between PVDF and first laminate ply but several layers underneath. The delamination surface is not consistent between two distinct plies and swaps between different interfaces varying between the second and the fifth laminate ply (Figure 5.2-3). It is remarkable that the delamination does not grow beyond the maximum crack length of approximately 26 mm. The delamination growth is arrested just at the end of the DSF area.

It can be stated that the Hybrid Bondline disbond arrest feature successfully worked in the fatigue test campaign. Even under unrealistic load levels for typical aerospace structures secure crack arresting could be achieved.

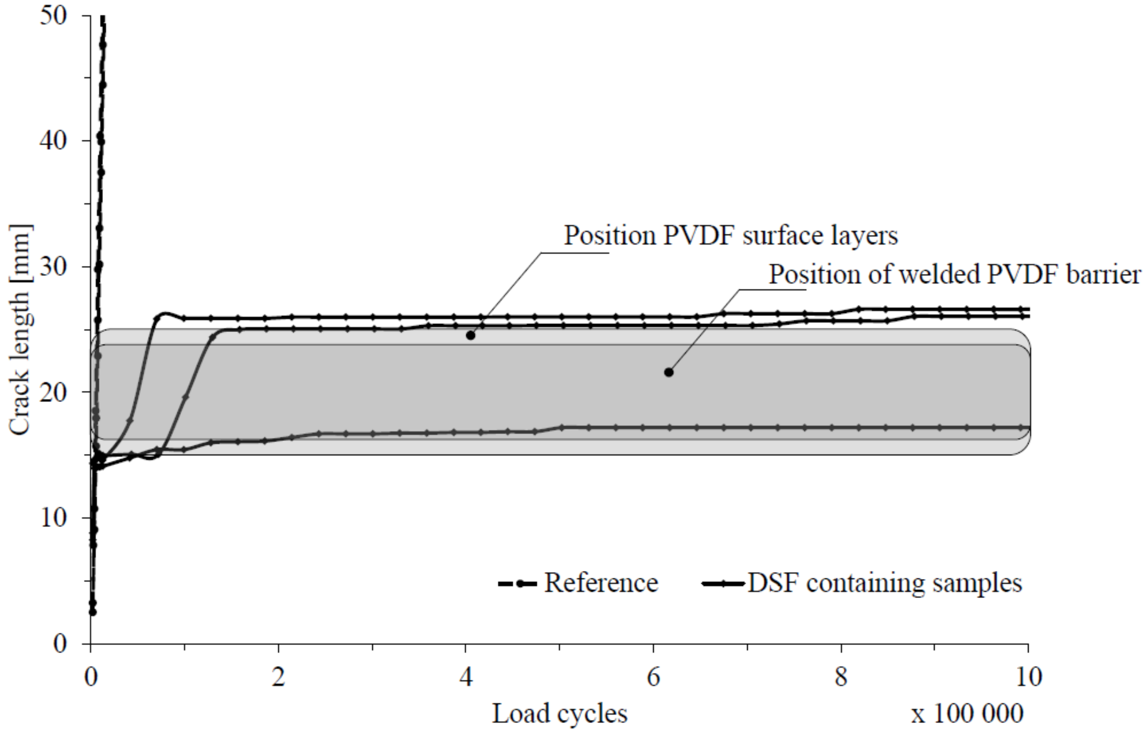

Figure 5.2-2: Maximum load 15.6 kN (5000 µm/m): Crack length versus load cycles for reference samples and DSF-containing samples

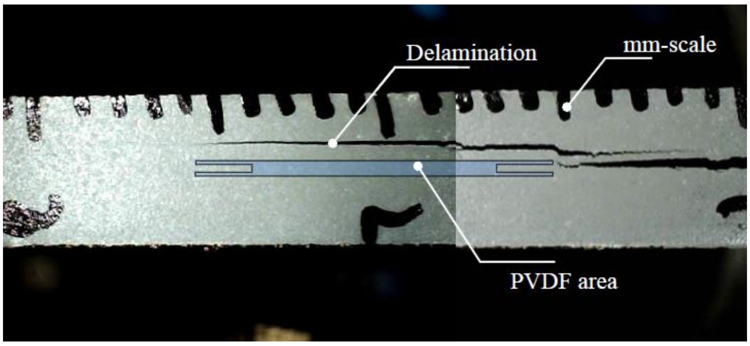

Figure 5.2-3: Microscopic image of crack opening under average load of a sample tested for 10^6 load cycles at a maximum load of 15.6 kN (5000 µm/m)

6.0 SUMMARY & OUTLOOK

The need for a disbond-stopping design feature is derived in this and previous work as one major step to enable certification of bonded primary composite aircraft structures. A novel crack-stopping design feature is developed in the European project BoPAcS that allows to safely stop growing cracks within bonded joints under fatigue loading. In contrast to existing approaches, neither additional faster elements, nor any kind of adhesive modifications are needed.

Two major tests are presented and discussed in this chapter to evaluate the crack-stopping capability of the novel concept: DCB tests and CLS tests. Both tests confirm the crack-stopping capability of the DSF concept. In DCB tests, crack growth is significantly retarded until ultimate breakage of the PVDF itself. In CLS tests, crack growth is fully stopped for 10^6 load cycles for various maximum loads without failure of the crack stopper. It is thus concluded that the working principle of the crack stopper is proven true.

The experimental work will be extended to the wide single lap shear (WSLS) test article based on results of single lap specimen not presented here. The WSLS configuration is a step towards the implementation of the crack stopper in high-loaded overlap joints, which are of high relevance to the aircraft industry. A non-linear FE analysis of both adhesive materials and a damage criteria of the adherends are required to calculate the load share within the joint and the strength of the joint more accurately. This is of high relevance for higher loads and hot/wet environmental conditions in which non-linear material behaviour becomes important to the joint designer.

During the investigations the origin of the good adhesion of the cocured PCDF-composite interphase was not finally found. This leaves room for concerns regarding a possible weak link. Further activities are planned in two directions. One is to investigate the sensitivity of the interphase to typical contaminants. The other one is looking into alternative materials which form a clear inter penetrating network.

6.1 Acknowledgments

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