EXPERIMENTAL INVESTIGATIONS OF THE DISBOND STOPPING CAPABILITY OF A NOVEL EPOXY-THERMOPLASTIC BONDLINE ARCHITECTURE FOR COMPOSITE JOINTS

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Abstract
A novel bondline architecture for bonded composite joints is presented. By implementing a distinct rather ductile thermoplastic phase, a physical barrier for growing disbonds is obtained and thus a more fail-safe design, respectively. Furthermore, the joint is established by using two different joining technologies, namely adhesive bonding and thermoset composite welding. A possible manufacturing technique is briefly described and assessed by means of Double Cantilever Beam (DCB) and Single Lap Shear (SLS) tests. The thermoplastic phase constrains disbonds by posing a physical barrier through a discrete change of materials. Thus, a significant increase against crack propagation is observed for DCB tests. However, visual examinations of the transition zone between both adhesive materials disclose sensitivity to a thickness mismatch between both materials leading to adhesion deficiencies in the vicinity of the thermoplast.

1. Introduction

1.1. Today's limitations of bonded composite joints

Adhesively bonded composite joints offer great advantages 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. Therefore, the development of adhesive bonds being capable for certification of primary aircraft structures is of high interest. However, due to certification requirements (see section 1.2) the implementation of bonded joints in aircraft structures is still limited to secondary parts or combined with so called "chicken rivets" if used in primary parts [1]. The main reasons for this limitation shall be briefly discussed.

The manufacturing process of structural bonded joints is influenced by many factors, e.g. surface treatment, adhesive curing cycle, curing conditions (e.g. pressure and temperature distribution), entrapped adherend's humidity and many more. Those factors may affect the long-term durability of the joint. Judging their impact on the joint's performance is complex and still subject of current research and scientific discussions.

In addition to manufacturing uncertainties, aging and fatigue life of bonded composite joints is still challenging to predict and also influenced by many factors (e.g. load level, strain rate

and environmental conditions) [2–4]. Thorough investigations of the interaction between those factors and their impact on the joint's long-term durability are hampered by the necessity of cost and time consuming experimental fatigue studies. Eventually, all those factors led to a significant scatter in the performance of bonded composite joints in the past with some working well and some failing after short time in service [5]. Those experiences have caused a distrust towards adhesive bonding as joining technology.

1.2. Certification requirements of bonded composite joints

Based on the experience in the past and the uncertainties mentioned above, the authorities, namely the Federal Aviation Administration (FAA, USA) and the European Aviation Safety Agency (EASA, Europe), specify two major prerequisites that have to be met to achieve certification of bonded composite joints for primary structures [6, 7].

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. Thus, influencing parameters and their tolerable deviations have to be determined. Despite a rigorous manufacturing quality management, one of the following methods has to be established to attain certification:

1. Disbonds greater 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. An NDI method that sheds light on the strength of adhesive bonds is currently not available. Porosities or voids may be detected by established methods like ultrasonic scanning or thermography. However, giving evidence that proper adhesion is achieved is not possible today.

In the end, a promising approach is to establish disbond stopping design features. Those must be developed and incorporated in each bond to prevent a possible disbond reaching a critical extent.

2. Novel approach of epoxy-thermoplastic bondline architecture

Many conventional epoxy adhesives for aeronautical applications are toughness-modified in order to reduce undesired brittle behavior that inherently applies for pure epoxy systems. However, adhesive toughening by incorporation of rubber or thermoplastic particles often leads to a degradation of stiffness and strength. Therefore, toughening is always a compromise between ductile behavior of good nature and stiffness or strength, respectively. The concept that shall be introduced here avoids the unfavorable trade-off by a strict separation of both functions (toughness and shear strength/stiffness for load transfer). This separation is achieved by dividing

the bondline into several areas as shown in [Figure 1]. On the one hand, a conventional high

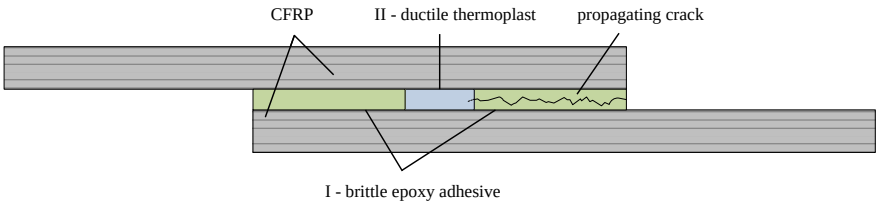

Figure 1. Principle alignment of both adhesives within the bondline

strength structural adhesive is used in phase I (Figure 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 by nature. Therefore, this physical barrier acts as disbond stopping feature within the bondline. Since the thermoplast is welded within the bondline (see section 3.1) not only a combination of material properties is achieved, but a combination of two different joining techniques: adhesive bonding and thermoplast welding (TCW). Thus, the joint does not solely rely on adhesive bonding which is accompanied with manufacturing uncertainties as discussed in section 1.1.

Due to its positioning in areas of low stresses, only minor stress concentrations are expected at the transition of both materials. Thus, the impact on the overall bond strength is expected to be small and tolerable. As shown in Figure 1 the disbond stopping thermoplastic phase could be integrated strip-wise perpendicular to the major load direction and crack path, respectively. For multi-loading cases or wide overlaps, a broad mesh-like application may be beneficial. For preliminary investigations a strip-wise alignment for the sake of simplicity shall be used, though.

3. Materials and Manufacturing

3.1. Materials

Specimens are manufactured using Hexply 8552/IM7 unidirectional prepreg material for adherends and Cycom FM 300K as common high strength film adhesive for aerospace applications. The adhesive thickness amounts to 0.2 mm for uncured conditions. For the thermoplastic phase a poly(venylideneflouride) (PVDF) film material of 0.125 mm thickness is chosen, due to its favorable properties. The suitability of PVDF material for welding of composite parts, also known as thermoset composite welding (TCW), was proven by Paton et al. [8]. Besides its ductile behavior (elongation at break of more than 50 %, yield strain of 7 %) and tensile strength of 50 MPa, the thermoplast has a melting temperature of 167° C.

The low melting temperature allows a certain manufacturing technique: The thermoplast shall be applied on both adherend parts. When the actual bonding is performed, the thermoplastic strips face each other. Due to the heat and pressure used for curing the adhesive, a welding between the facing strips of PVDF can be achieved.

3.2. General Manufacturing Details

The performance of the novel joining concept shall be assessed by mechanical testing. For this purpose, Single Lap Shear (SLS) and Double Cantilever Beam (DCB) specimens are chosen. Purely bonded samples (without thermoplastic strip) are used as reference. The DCB specimens are manufactured and tested for determination of the fracture toughness under pure mode I loading conditions. Thus, those tests are suitable for a first evaluation of the disbond stopping capability. SLS tests are conducted in order to determine the influence of the thermoplastic strip on the static strength. A set of five specimens is manufactured for every configuration. For all samples containing a thermoplastic strip, PVDF of 5 mm in width is used.

A peel ply (Hexforce T0098) is applied to all prepreg surfaces to ensure a constant overall surface finish. All bonding surfaces are ground with sandpaper of 500 grit and cleaned with acetone prior to bonding to obtain sufficient adhesion.

The joint manufacturing can be divided into three steps as shown in Figure 2. The thermoplastic strips are put in place before curing of the adherend parts. The curing cycle is conducted as specified by the material data sheet. Since the thermoplast's melting point matches the curing temperature of the CFRP, a strong bond is realized between the thermoplast and the composite's matrix system. The thermoplastic strips are placed on both adherend surfaces. Thus, the strips face each other when both parts are joined. In a second step, the actual bonding is conducted

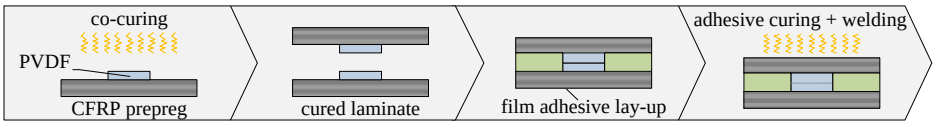

Figure 2. Manufacturing steps for hybrid (bonded & welded) joint

by placing the epoxy adhesive on one adherend with the area of thermoplastic strips left open.

In the final step, the adhesive is cured and the bond is thus established. Both the adhesive curing and the thermoplastic welding shall be performed simultaneously. The applied curing temperature of 180° C and pressure of 1.5 bar ensures a proper weld at the interface of both PVDF layers. Pressure and temperature are maintained for 2 hours to allow a complete polymerization of the epoxy adhesive.

3.3. Manufacturing of Double Cantilever Beam Samples

The specimens are manufactured according to ISO 15024 test standard. The adherends are laid in a uni-directional manner containing twelve 0°-plies orientated in longitudinal direction. With a ply thickness of 0.125 mm the overall adherend thickness amounts to 1.5 mm.

Two types of specimens are manufactured for DCB tests: purely bonded samples using the epoxy film adhesive and samples containing both, the PVDF strip at one distinct position surrounded by the conventional film adhesive. The latter give an indication of the influence of the

thermoplastic strip on a growing disbond. According to the appropriate position for the disbond stopping feature determined in section 4.1, PVDF strips are inserted 42 mm from the loading point of the piano hinges (Figure 3). One PVDF containing sample is also examined by the use of Gom Aramis system which allows a contact-free measurement of the strain field. The Aramis system is used to investigate the crack propagation edgewise which allows strain measurements of the bondline.

3.4. Manufacturing of Single Lap Shear Samples

The SLS-samples are built in wide accordance to ASTM D 1002 test standard. The plates consist of 16 plies $[+45/0/-45/90/+45/0/-45/90]_s$ leading to a plate thickness of 2 mm. In contrast to the test standard, the overlap length is set to 25 mm to allow investigations of bonded and welded joints. Purely bonded specimens using FM 300 film adhesive are produced as reference. Specimens containing both, the epoxy adhesive and the thermoplast, are manufactured to determine the influence of the thermoplastic strip on the overall joint tensile strength. For this purpose, the PVDF strip is placed centrally in the overlap area. Hence, additional stress peaks are expected to be of minor impact due to low stress concentrations in the center of the overlap.

4. Results

4.1. Double Cantilever Beam Results

All tests are carried out displacement driven with a testing speed of 0.5 mm per minute to minimize undesired abrupt crack growth of large extent. Crack growth is recorded by use of a traveling microscope. The critical energy release rate G_{IC} is calculated according to area method as given in [9].

$$G_{IC} = \frac{1}{2 \cdot b \cdot \Delta a} \cdot (P_1 \cdot \delta_2 - P_2 \cdot \delta_1) \tag{1}$$

The parameters needed for calculating G_{IC} are specimen's width b, load P_1, cross head displacement δ_1 at crack length a. The load then decreases to P_2 and the displacement increases to δ_2 whilst the crack length grow to $a + \Delta a$.

Due to the adhesive's brittle nature, the contour of the cross head displacement versus load plot lead to jagged curves for the purely bonded specimens. This stick-slip effect causes diverse crack propagation behavior for the five specimens tested. Cracks appear to grow rather abruptly which is characterized by a spontaneous load drop after linear load increase. The fracture pattern reveals a mixture of different failure modes since adhesive, cohesive and light fiber tear failure are observed. For the reference samples it was decided to evaluate the critical energy release rate (ERR) by using the crack length of the first measurable crack arrest point. The average ultimate load amounts to 110 N which lead to a value of 733 J/m^2 for G_{IC}. The average cross head displacement for first crack amounts to 7.0 mm.

As standard deviations vary significantly in all parameters (load, cross head displacement and crack length) at increasing numbers of cracks, the crack stopping thermoplast should be inserted

at a rather short crack length, preferably between initial crack (pre-loading) and first crack, thus constraining its propagation measurably. The average length for the initial crack amounts to $\mu = 36.52\,\text{mm}$ with a relative standard deviation σ of 2.42 mm assuming that the crack length is normally distributed. The range of the disbond stopping feature location should begin just after a 2σ interval from the initial crack as shown in Figure 3: $\mu + 2\sigma \approx 42\,mm$. Thereby, the occurrence of initial cracks in that range can be precluded by 95.4 %.

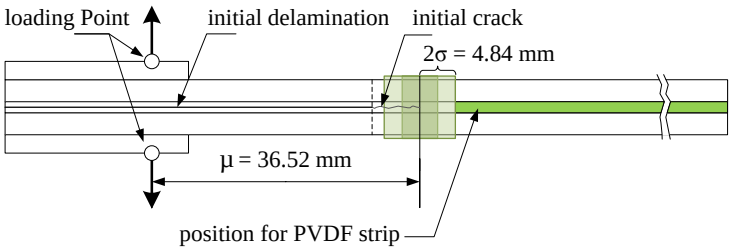

Figure 3. Chosen position for PVDF strip in DCB samples based on reference results

For the specimens containing one PVDF strip, ultimate load for the first crack (ahead of the PVDF strip) reaches an average of 42.9 N and deviates by 31.4 % which is an indication of poor adhesion quality in the vicinity of the thermoplastic strip. In fact, the fracture pattern reveals adhesion failure of the epoxy phase ahead of the PVDF strip. However, the slow and stable crack growth is stopped at the transition to the thermoplastic phase. Both, cross head displacement and applied load increase significantly before the crack grows spontaneously and unstable through the PVDF and into the epoxy phase again. An average of 172.4 N for ultimate load and average of 13.2 mm cross head displacement is obtained. In fact, Aramis measurements reveal a compression loaded area in front of the crack tip due to high beam deformations (Figure 4). After cohesive failure of the PVDF the crack grows extensively through the composite adhesive interface indicating again poor adhesion of the epoxy. Arrest occurs when the beam lost sufficient kinetic energy and thus the remaining energy equals the critical energy release rate ahead of the crack tip. Hence, using the measured crack length for calculation of the G_{IC}-value gives a misleading mean value of very strong crack constraining of the PVDF and very poor energy absorption of the poorly bonded epoxy. Nevertheless, the averaged fracture toughness is calculated to 1165 J/m^2 which is still 59 % above the reference (Figure 5) .

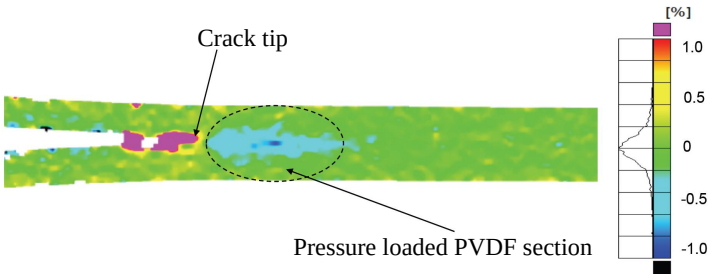

Figure 4. ARAMIS evaluation (strain in peel direction) of loaded DCB sample containing PVDF strip prior failure of the PVDF strip

4.2. Single Lap Shear Results

The purely bonded reference samples show a homogeneous course of testing with minor scatter. The average ultimate load amounts to 10.4 kN with a deviation of 2.8 %. In addition, the global failure strain level is determined to 0.35 % by the use of strain gauges. For all specimens, fracture patterns are characterized by a share of about 40 % of cohesive failure with a simultaneous delamination of the first ply beneath the lap-bond area (first ply failure).

PVDF strips within the adhesive layer are found to have significant impact on the failure behavior. Thus, specimens are observed to fail rather continuously than abruptly. However, a mere ultimate average load of 43 % of the reference value is reached. The fracture patterns reveal again adhesion failure of the film adhesive and cohesive failure for the thermoplast as seen with the DCB samples. Unlike single phase specimens, the load curve shows an unique course which is characterized by two stages of failure as illustrated in Figure 5.

5. Discussion

Although adhesion deficiencies in the vicinity of the PVDF material is observed, the proposed working principle is proven true. The unambiguous increase of load (56 %) prior to crack growth in DCB tests compared to reference results confirms the crack constraining capabilities of the distinct ductile thermoplastic phase.

For SLS tests, it is shown that no abrupt load drop occurs once peak load is reached. Instead, a rather ductile failure is observed which can be concluded from flattening of load decrease after peak load. Furthermore, it can be concluded that the major share of the tensile load is in fact carried by the PVDF phase which is well-welded and therefore shows good cohesive and adhesive strength. However, the appearance of a weak bond close to the PVDF decrease the ultimate load significantly.

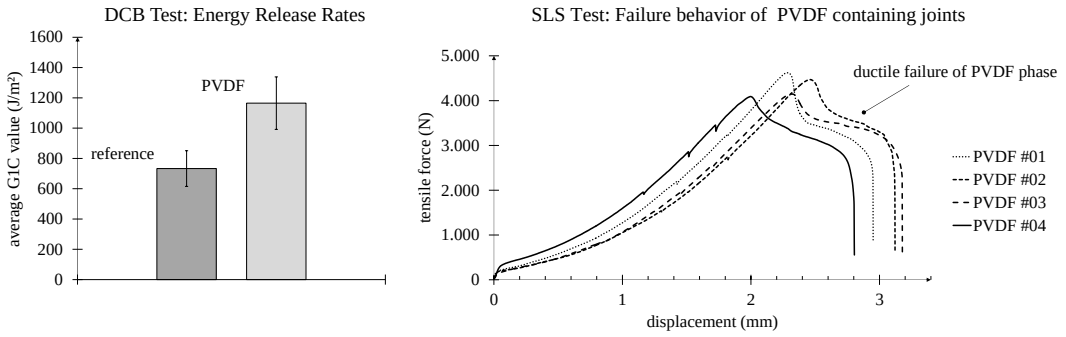

Figure 5. Test results: left - comparison of energy release rates for reference and PVDF samples; right - load-displacement results for SLS tests of PVDF samples

The PVDF's influence on bonding quality is believed to be caused by an post-curing thickness mismatch. The thickness after curing amounts to merely 0.13-0.15 mm for the FM 300 whereas the PVDF keeps its thickness of 0.2 mm. Hence, subsequent studies focus on solving those technological issues. It is proposed to vary the thickness of both adhesive systems to allow simultaneous bonding and welding. Furthermore, decoupling both joining processes (bonding

and welding) by the use of highly localized ultrasonic welding is considered to overcome the challenges identified within this study.

6. Conclusion

The proposed bonding concept is a promising approach to overcome certification issues for bonding of composite aircraft structures. A fail-safe joint could be achieved by applying discrete ductile phases to subdivide the bondline into independent areas. Besides, combining two different joining technologies offers a joint that not solely relies on the quality of the adhesive bond which is accompanied with manufacturing uncertainties. Further work will focus on the identified adhesion issues in the vicinity of the thermoplastic phase.

7. Acknowledgments

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