DIARC PLASMA COATING FOR RELIABLE AND DURABLE STRUCTURAL BONDING OF METALS

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Abstract

DIARC vacuum plasma surface treatment was tested for comparing its performance in aluminium, titanium and steel structural epoxy bonding with existing methods. Static single lap shear and static double lap shear specimens were tested at room temperature as dry and wet. Wedge tests were also performed in different environments. DIARC coating provided good results with all metals in all testing. In a case study the coating applied to sandwich honeycomb steel inserts improved their torque strength over 100 %.

1 Introduction

The lack of robust and highly reliable surface preparation techniques has limited the use of bonded joints in highly loaded metal to metal and metal to composite structural joints in aircraft applications. With aluminum and titanium the grit blast AC-130 Sol-Gel process can provide acceptable results, but the technique requires a competent mechanic following strict process instructions without any deviations. Also, in aircraft applications the process usually requires chromate primers, which induces a healthy risk. For steel there has not been an acceptable method available that would satisfy aircraft quality criteria for structural bonding.

The objective of this study was to investigate the performance of new DIARC coating method for metal bonding. The acronym DIARC refers in this study to the registered trade mark DIARC®, whose products are e.g. amorphous diamond coatings for tools and components and nanocomposite coatings for functional applications [1]. The results were compared to grit blast silane (GBS) and grit blast AC-130 Sol-Gel (SG) testing. Typical aircraft grade aluminium and titanium along with stainless and high strength steels were selected for testing.

A case study in this investigation was a bonded steel insert in composite sandwich panels. The inserts provide attachment points for other structures or installations. A potted insert often fails from the interface between the insert and adhesive potting compound with too small torque load. Since the replacement of the insert is laborious, an improvement for the adhesion was searched with the DIARC coating.

2 Materials and processes

2.1 DIARC coating method

DIARC process is a novel method for adhesive bonding of dissimilar materials. In the DIARC process the metal part is plasma treated in a vacuum chamber below 100 °C temperature. Ions with enough kinetic energy form a thin (from nanometers to microns) well adherent amorphous nanostructured layer when they hit the surface. The surface layer is dense, hard, corrosion resistant, has very low coefficient of friction, and is also bondable to epoxy. In addition to epoxy bonding, the DIARC process can be used for coating tools, sliding parts and molds for lower friction and wear resistance, and for replacing other environmentally hazardous corrosion protection coatings on metals (e.g. epoxy primers containing chromates).

For epoxy bonding the treated metal part does not need any additional primer. It also stays stable and is robust to the bonding process. The DIARC pretreatment thus decreases the workload in the final bonding and also decreases the requirements for the surface treatment stage of the process. Also the DIARC process does not involve any harmful substances or chemical processes.

2.2 Silane based surface treatments

Silane surface treatment methods are based on γ-glycidoxypropyltrimethoxysilane, which creates a chemical bond with the fresh and still active aluminium oxide layer. In the other end of the hydrocarbon chain the silane has an epoxy group which is bondable to epoxy adhesives or primers.

In the grit blast silane (GBS) processes the metal surface must be cleaned with a solvent and grit-blasted no longer than 5 minutes before applying the 1 % silane solution. The silane must be cured at 110 °C for one hour. After silane treatment the surface must be bonded or covered with a primer within 2 hours.

In the Sol-Gel AC-130 method small amounts of glacial acetic acid (GAA) and zirconium n-propaxine (TPOZ) are added to a 1 % silane-water solution. These chemicals lower the pH level of the solution from approximately 5.3 to 3.5.

AC-130 is manufactured in 4-component kits and in 2-component kits. Some older results presented in this paper were achieved using the 4-component kit. In most recent tests described in this paper 2-component kits AC-130-2 were used. Both kit types should provide the same performance [2].

The grit blast AC-130 Sol-Gel process is slightly more robust than the GBS method. After cleaning and grit blasting the solution should be applied on the surface within 30 minutes. The Sol-Gel is cured at room temperature for one hour. The surface must be bonded or covered with a primer within 24 hours.

White pure 180/220 grit aluminium oxide was used in grit blasting. The blowing agent was clean air. The blasting pressures used for aluminium, titanium and steels were 3, 4.5 and 6 bars, respectively.

An intention in this investigation was also to avoid chromate primers. However, in some cases the use of primer was found necessary in order to achieve a durable bond. The BR 6747-1 primer was used with grit blast AC-130 Sol-Gel treatment in some cases. The primer was applied using a foam brush and cured in on oven 1 hour at 120 °C. The target primer thickness was 2.5 –7.5 µm.

In stainless steel single lap shear reference testing the grit blast Sol-gel with the primer application was used. The reference wedge tests for stainless steel presented in this paper were accomplished with the grit blast Sol-Gel method without primer. The wedge test reference values for titanium were achieved with the grit blast AC-130 Sol-Gel treatment followed by the BR 6747-1 primer application.

2.3 Metals

Aircraft grade 7075-T6 bare and clad aluminiums were used in static single lap and double lap shear testing and in wedge testing. Clad was not removed from the specimens prior to surface treatment and bonding.

Titanium 6Al-4V and AISI 304 stainless steel were used in static single lap and double lap shear testing and in wedge testing, High strength AISI 4130N steel specimens were also tested with wedge testing.

2.4 Adhesive film

FM300-2 film was used as an adhesive in most specimens. The epoxy adhesive film had the areal weight of 490 g/m^2. The adhesive film was cured 2 hours in an oven at 120 °C under 0.7 bar vacuum pressure.

2.5 Case study

2.5.1 Steel inserts

Inserts used in the case study were made of passivated stainless steel 1.4301 DIN 17440. The composition of steel is equivalent to AISI 304. The insert had 11.35 mm flange diameter and M5 threads.

2.5.2 Composite panels

Steel inserts were installed into carbon laminate faced sandwich panels with Nomex-honeycomb core. Two sets of panels were used. Both panels had approximately 1 mm face sheets made of carbon/epoxy fabric prepreg and 48 kg/m^3 density Nomex-core. In the first panel the core height was 10 mm and in the second panel the core height was 12 mm. The core was bonded with FM300K adhesive film. The sandwich was co-cured in one stage in an autoclave.

2.5.3 Potting compound

In insert installation a potting material made of EA9396 epoxy resin and 10 w. -% phenolic microballoons was used. This material was also tested with static single lap shear specimens in order to compare the DIARC method with existing steel surface preparation methods. The potting material was cured in room temperature overnight and post-cured 2 hours at 60 °C.

3 Testing methods

3.1 Single lap shear specimens

The single lap shear specimens were prepared according to ASTM 1002 guidelines. The bondline length was 12.7 mm (0.5 inch). The thickness of steel and titanium specimens was 2 mm. The aluminium specimens were made out of 3.2 mm thick plates. The specimens were tested in static tension as room temperature dry (RTD) and also at room temperature after 30 days exposure in the hot wet environment 60 °C / 98 % RH (RTW).

3.2 Double lap shear specimens

Double lap shear specimens had 38.5 mm (1.5 inch) overlap length. The center adherends had the same thickness as the single lap shear specimens while the thickness of the outer straps was half of that (see Fig. 6 and 7). The specimens were tension tested at RT/Dry and RT/Wet after 30 days exposure in the hot wet environment 60 °C / 98 % RH.

3.3 Wedge tests

ASTM D3762 wedge testing was used to measure the bonding durability of the surface preparations. Each set contained 5 specimens. In this study the following acceptance values were used for the bond with the FM300-2 film: initial crack length less than 45 mm, crack growth in 48 hours less than 6.5 mm and failure mode more than 80 % cohesive.

ASTM D3762 advises to use 3.2 mm thick aluminium plates with 3.2 thick wedges. These thicknesses were used in the study.

Crack opening energy G_I for alumiunium specimens was calculated using Equation (1) [3], in which Y is the thickness of the wedge, h is the thickness of the plate, E is the Young's modulus of the material and a is the crack length. The plate and wedge thicknesses for steel and titanium specimens were selected in order to have a reasonably close match with the G_I value of the aluminium specimens. With 2 mm thick titanium plates 4.9 mm thick wedges were used and with 2 mm thick steels plates 3.2 mm wedges were used.

$$G_I = \frac{Y^2 E h^3 \left[3(a+0.6h)^2 + h^2\right]}{16\left[(a+0.6h)^3 + ah^2\right]^2} \qquad (1)$$

The crack opening energy with steel specimens was lower than with aluminium specimens [4]. It did not, however, affect too much the crack growth rate or the failure mode, which is also a very important indicator of the durability.

Wedge testing was started using a typical accelerated hot wet aging environment 60 °C / 98 % RH. More demanding exposure conditions were created by immersing the specimens in hot (60 °C) tap and salt water. The North Baltic Sea water with 0.5 % salt content was used. The pH in the fresh water tank was 9.9 and in the salt water tank it was 8.9. The Redox-potentials in fresh water and salt water tanks were, respectively, -140 mV and -96 mV.

Another demanding environment used in the tests was neutral salt fog at 35 °C. These tests followed the recommendations of the ASTM B 117 standard.

3.4 Case study: Insert testing

Blind inserts with an installation collar were installed into carbon laminate faced sandwich panels. An M5 steel bolt with a washer was installed in to the inserts and the inserts were torque loaded with an electric torque wrench to ultimate load.

The tests were performed with unexposed and exposed specimens at room temperature. The exposure was 56 days at 60 °C / 98 % RH.

4 Results

4.1 Single lap shear testing

Average single lap shear strength (force divided by joint area) results of DIARC treated stainless steel, titanium and bare and clad aluminiums are shown in Fig. 1. Specimens were tested at room temperature as dry and after 30 days moisture exposure at 60 °C / 98 % RH. From 6 to 12 specimens were tested in each set.

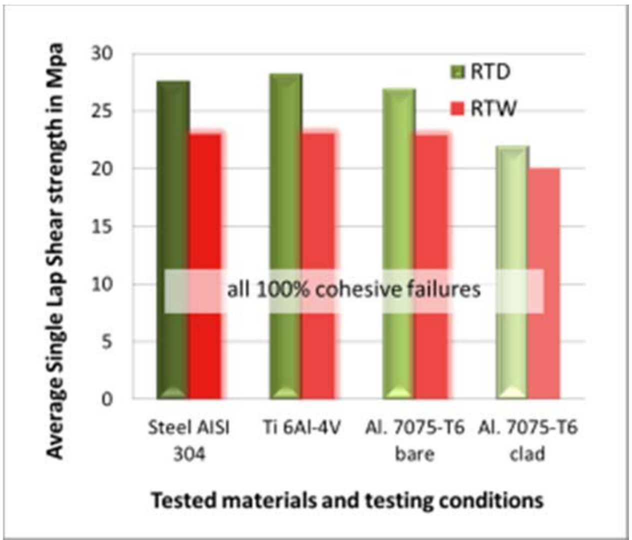

Fig. 1. Measured average shear strength of stainless steel, titanium and bare and clad aluminium single lap shear specimens with DIARC treatment, tested at room temperature as dry (RTD) and wet (RTW).

Average room temperature dry shear strength values for steel, titanium and bare aluminium were all above 25 MPa, which is a typical quality control acceptance value for a dry FM300-2 adhesive bond cured in a vacuum bag. The average shear strength of dry clad aluminium specimens was in these tests 22 MPa. The room temperature wet single lap shear values for steel, titanium, bare aluminium and clad aluminium were 17 %, 18 %, 15 % and 9 % lower, respectively.

The failure modes of all dry and wet specimens were 100% cohesive. A typical failure surface is shown in Fig. 2.

Fig. 2. Typical cohesive failure mode of stainless steel AISI 304 (SS) single lap shear specimens with DIARC treatment and FM300-2 adhesive film tested at room temperature as wet (RTW).

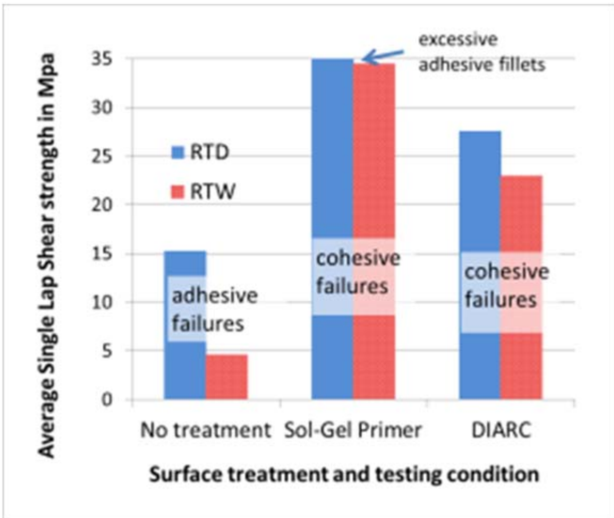

Fig. 3. Measured average shear strength of stainless steel AISI 304 single lap shear specimens with no treatment, grit blast Sol-Gel primer treatment and DIARC treatment, bonded with FM300-2 and tested at room temperature as dry (RTD) and wet (RTW).

Average shear strength values measured with different treatments of stainless steel single lap shear specimens are shown in Fig. 3. The first set was bonded on the passivated AISI 304 surface without any treatment. The second treatment was grit blast Sol-Gel with a primer. The third method was the DIARC coating. The DIARC treatment was tested with 12 specimens in both sets while the other methods were tested with 4 specimens in each set.

The results in Fig. 3 show that the specimens without any surface treatment had low shear strengths and mostly adhesive failure modes as dry and wet. The grit blast Sol-Gel primer treatment performed well in these tests as compared to the DIARC treatment. Both treatments resulted also in cohesive failure modes.

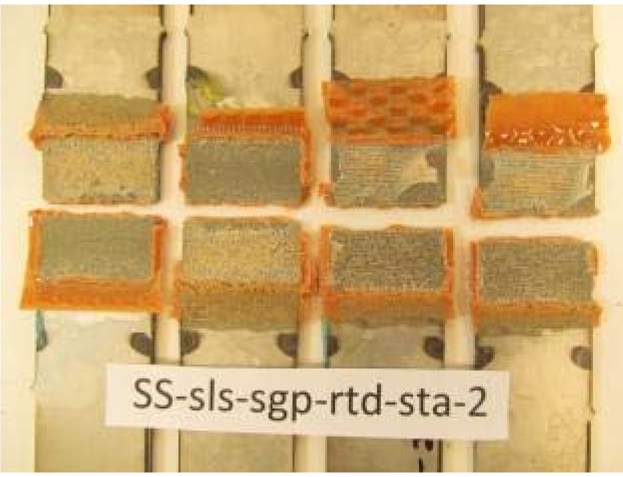

Fig. 4. Typical cohesive failure mode of stainless steel AISI 304 (SS) single lap shear specimens with grit blast Sol-Gel primer treatment tested at room temperate as wet (RTW). The excessive fillets formed during the bonding with FM300-2 are visible.

The high shear strength values in Fig. 3 for single lap shear specimens, measured with the grit blast Sol-Gel primer, may have limited importance since the value is highly affected by the size and shape of the adhesive fillet formed during the bonding process. The high values gained with the SGP-method may partly be explained with excessive fillets shown in Fig. 4 when compared with the normal fillets of the specimens with the DIARC treatment (Fig. 2).

4.2 Double lap shear testing

Static tension test results of double lap shear specimens with the DIARC treatment are shown in Fig. 5. The average strength (force divided by joint area) has been given in the figure in order to compare the results. Steel and titanium were tested with 6 specimens while aluminium was tested with four specimens.

Stainless steel had 11 MPa average shear strength as dry and slightly lower strength 10.5 MPa as wet. Titanium had average values of 25 and 23 MPa as dry and wet, respectively. Bare aluminium had the same average shear strength value 21.4 MPa as dry and wet. Clad aluminium had average shear strength 20.9 MPa as dry and 20.5 MPa as wet.

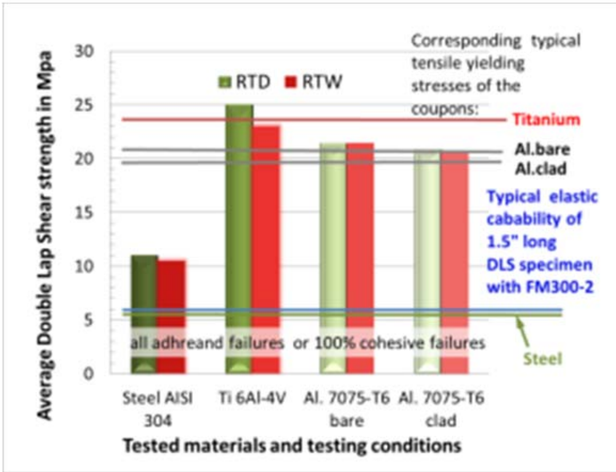

Fig. 5. Measured average strength of 1.5” long double lap shear specimens with the DIARC treatment and FM300-2 adhesive film tested at room temperate as dry (RTD) and wet (RTW). Additional horizontal lines represent corresponding MIL-HDBK-5 tensile yielding stresses of the materials in tested DLS coupons.

All stainless steel specimens failed cohesively in the bondline. The failure modes with other metals were mixed. Some specimens failed due to the adherend failure and some specimens failed cohesively in the bondline. Typical failure modes are shown in Fig. 6 and 7. Two of titanium specimens failed due the adherend failure and two failed cohesively in the bondline (Fig. 7). Most of the aluminium specimens had adherend failure mode, which explains that measured strengths of dry and wet specimens were almost the same.

Stainless steel specimens had in these tests much lower measured strength than the other specimens. This can be explained with the lower yielding stress of the material. With the used forces and configuration the stainless steel straps start to yield very early. The yielding occurs before the typical FM300-2 elastic shear stress capability of the joint is reached. The strap yielding increases remarkably the adhesive film plasticity in the ends of the joint and thus the ultimate strength is low [5]. Fig. 6 shows the typical plastic deformation of the tested stainless steel specimens.

Fig. 6. Typical plastic deformation and cohesive failure mode in the doubler ends of stainless steel AISI 304 (SS) double lap shear specimens.

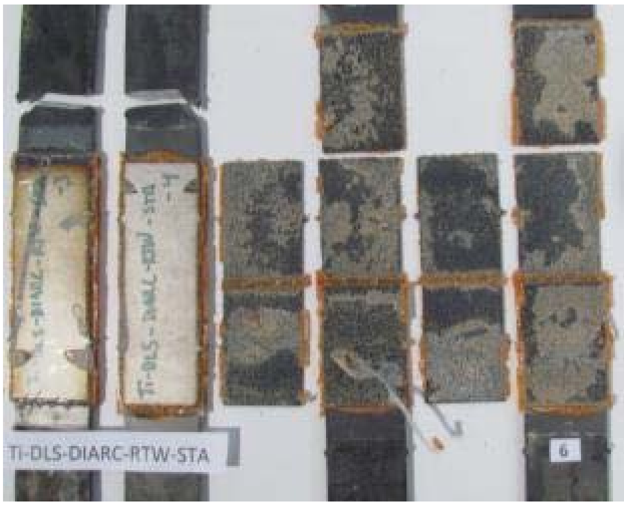

Fig. 7. Typical failure modes of titanium double lap shear specimens with the DIARC treatment. Two specimens had adherend failure modes while two specimens failed cohesively in the bondline.

The additional horizontal blue line drawn in Fig. 5 represents the typical elastic capability of 1.5 inch long FM300-2 double lap joint. The other lines represent force levels where yielding of the materials in the used joint configuration starts. The yielding stresses have been obtained from MIL-HDBK-5.

It can be seen that the tensile yielding stress level of steel straps is slightly lower than the yielding shear stress of the adhesive. The yielding stress values are higher with titanium and aluminium specimens and thus full elastic and plastic capability of the adhesive can be reached. This also may lead to the adherend failure mode as seen in Fig. 7.

4.3 Wedge testing at hot wet environment

4.3.1 Steel specimens

Average crack growth values measured in hot/wet wedge tests of steels are shown in Fig. 8. All AISI 304 specimens had small initial crack lengths comparable to the lower G_I values. The average value was 30 mm. The average initial crack lengths of each set tested at hot/wet and in immersion are given in Ref. [3]. The crack growth with the DIARC treatment was less than 5 mm in 48 hours. The failure mode, as shown in Fig. 9, was 95% cohesive.

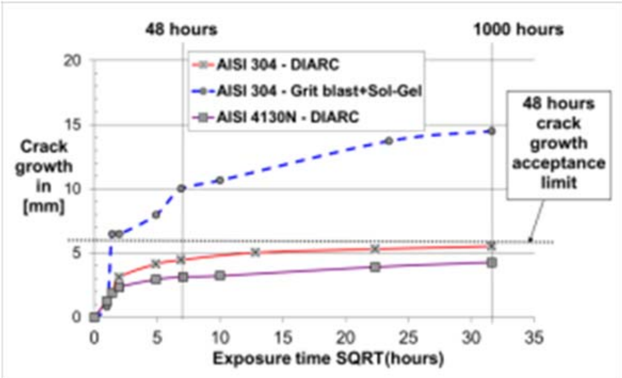

Fig. 8. Crack growth of stainless steel AISI 304 and high strength steel AISI 4130N wedge test coupons with grit-blast AC-130 Sol-Gel and DIARC surface treatment at hot wet environment 60 °C / 98 % RH.

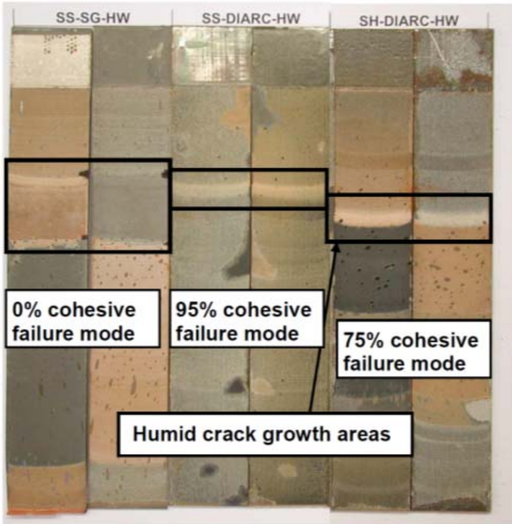

Fig. 9. Typical failure modes of stainless steel AISI 304 (SS) and high strength steel AISI 4130N (SH) wedge test coupons prepared with grit blast Sol-Gel (SG) and DIARC treatments after 1000 hours exposure at hot wet environment 60 °C / 98 % RH.

The reference treatment for steel was grit blast Sol-Gel. For these specimens the initial crack length was still small, but the crack growth in 48 hours was 10 mm which exceeded the acceptance limit 6 mm. Also, the failure mode was totally adhesive (Fig. 9).

The DIARC coated high strength steel AISI 4130N specimens had average initial crack length 38 mm. The crack growth and failure modes were at acceptable level.

4.3.2 Titanium specimens

Average crack growth values measured in hot/wet wedge tests of titanium are shown in Fig. 10. For titanium the reference surface treatment for DIARC was grit blast and Sol-Gel followed by the BR 6747-1 primer (SGP) application. SGP was selected since from our earlier experience [4] the Sol-Gel without primer did not give acceptable results for titanium.

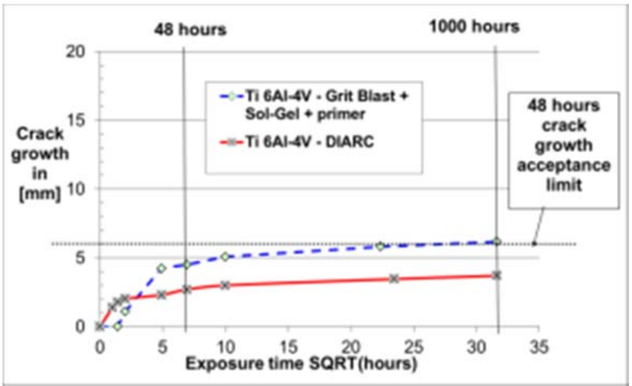

Fig. 10. Crack growth of titanium Ti 6Al-4V wedge test coupons with grit-blast AC-130 Sol-Gel primer (SGP) and DIARC surface treatment at hot wet environment 60 °C / 98 % RH.

The results in Figures 10 and 11 show that for titanium the DIARC and SGP treatments both gave acceptable results. Average initial crack lengths of the specimens were 41 mm and 37 mm for DIARC and SGP specimens, respectively. The average crack growth for the DIARC treated titanium specimens was 2.7 mm in 48 hours and the failure mode was 90 % cohesive. For SGP specimens the average crack growth in 48 hours was 4.5 mm and the failure mode was 95 % cohesive. Typical failure modes can be seen in Fig. 11.

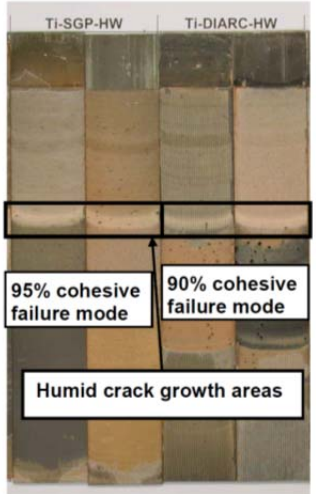

Fig. 11. Typical failure modes of titanium (Ti) wedge test coupons prepared with grit blast Sol-Gel primer (SGP) and DIARC treatments after 1000 hours exposure at hot wet environment 60 °C / 98 % RH.

4.3.3 Aluminium specimens

Average crack growth values measured in hot/wet wedge tests of aluminiums are shown in Fig. 12. For bare 7075-T6 aluminum the reference surface treatment for DIARC was grit blast silane (GBS). The GBS method was selected since in our earlier tests [4] the grit blast Sol-Gel without a primer did not give acceptable results for bare aluminium. However, with clad 7075-T6 aluminium the grit blast Sol-Gel method worked well even without primer.

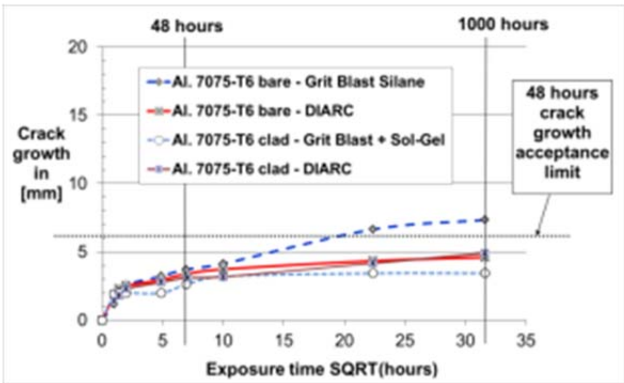

Fig. 12. Crack growth of bare and clad 7075-T76 aluminium wedge test coupons with grit blast silane, grit blast AC-130 Sol-Gel primer (SGP) and DIARC surface treatment at hot wet environment 60 °C / 98 % RH.

The results in Figures 12 and 13 show that for aluminiums all treatments gave acceptable crack growth values.

Initial crack lengths of all specimens were below 45 mm and the failure modes were more than 80 % cohesive. Typical failure modes of the specimens can be seen in Figure 13.

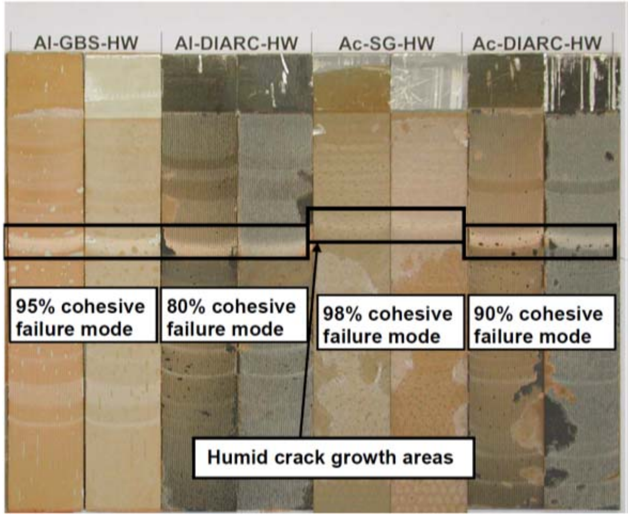

Fig. 13. Typical failure modes of bare and clad aluminium (Al and Ac respectively) specimens prepared with GBS, SG and DIARC treatments after 1000 hours exposure at hot wet environment 60 °C / 98 % RH.

4.4 Wedge testing in immersion

4.4.1 Steel ant titanium specimens

Average crack growth values measured in immersion wedge tests of steel and titanium specimens are shown in Figures 14 and 15. These tests were performed only with DIARC coated specimens.

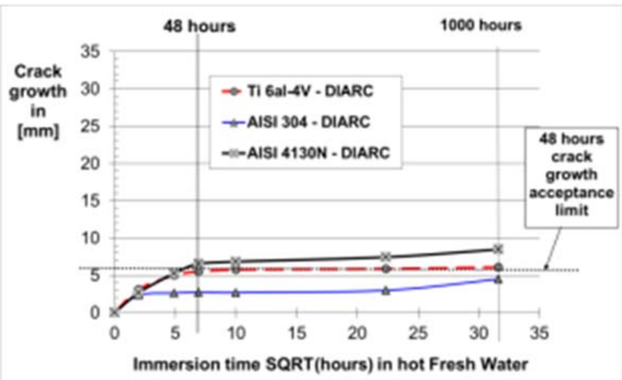

Fig. 14. Crack growth of titanium, stainless steel AISI 304 and high strength steel AISI 4130N wedge test specimens after DIARC surface treatment method, hot (60 °C) fresh water immersion.

All specimens had acceptable initial crack length. In fresh water immersion (Fig. 14) the crack growths of titanium and stainless steel specimens were below the acceptance limit 6 mm in 48 hours. The high strength steel behaved slightly differently and had 6.6 mm average crack growth.

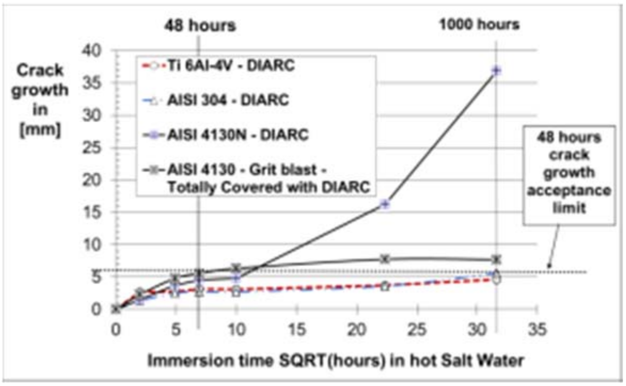

Fig. 15. Crack growth of titanium, stainless steel AISI 304 and high strength steel AISI 4130N wedge test specimens after DIARC surface treatment method, hot (60 °C) salt water immersion.

The failure mode for titanium in fresh water was 100 % cohesive (Fig. 16). The stainless steel had 70 % cohesive failure (Fig. 16).

The high strength steel corroded in immersion from the uncoated edges of the specimens. The corrosion advanced between the parent material and coating. The failure mode of the high strength steel in hot fresh water immersion was 85 % cohesive in the bondline and 15 % adhesive between the parent material and coating, as shown in Fig.17.

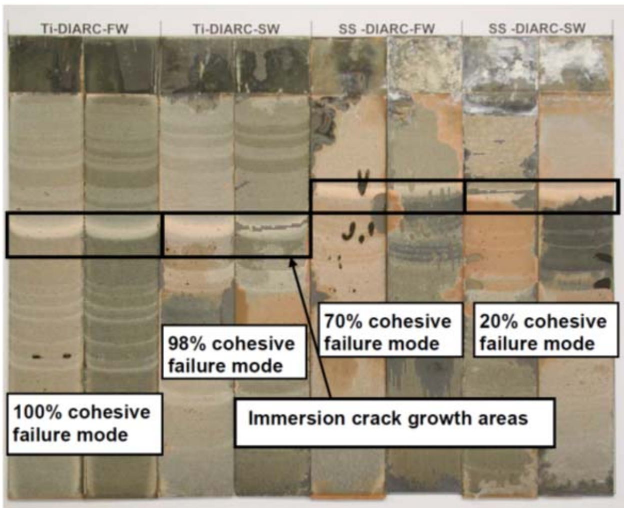

Fig. 16. Typical failure modes of titanium and stainless steel AISI 304 (SS) prepared with DIARC treatment after 1000 hours immersion at hot (60 °C) fresh and salt water.

In 0.5 % salt water immersion (Fig. 15) the crack growth of all specimens in 48 hours was below the acceptance limit 6 mm. After 100 hours the advanced edge corrosion of the high strength steel material increased the crack growth rate remarkably for the uncoated edge specimens.

The failure mode for titanium in salt water was 98 % cohesive (Fig. 16). The failure mode for stainless steel was only 20 % cohesive (Fig. 16).

High strength steel with uncoated edges corroded significantly in salt water. The average failure mode of the uncoated edge high strength steel coupons was 5 % cohesive in the bondline and 95 % adhesive between the parent material and coating, as shown in Fig.17.

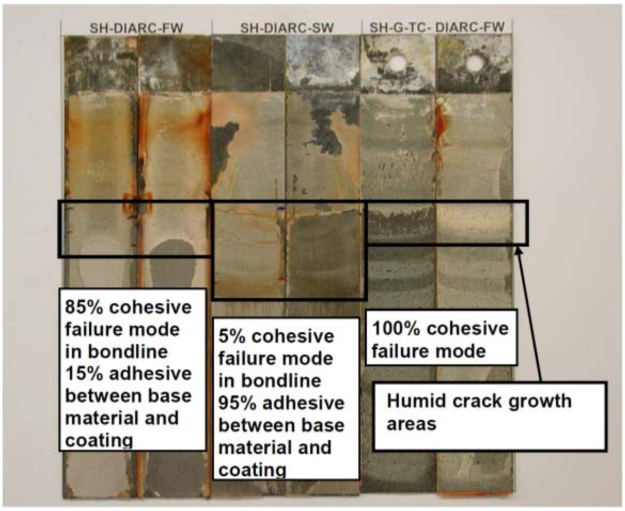

Fig. 17. Typical failure modes of titanium and stainless steel AISI 304 (SS) and high strength steel AISI 4130N (SH) prepared with DIARC treatments after 1000 hours immersion at hot (60 °C) fresh and salt water.

In the second test series the high strength steel coupons were cut in advance and individually grit blasted and within 5 days coated from all edges with the DIARC treatment. This approach improved the performance remarkably. The crack growth (Fig. 15) was gradual and the failure mode was 100 % cohesive (Fig 17).

4.4.2 Aluminium specimens

Average crack growth values measured in immersion wedge tests of bare and clad aluminium specimens are shown in Figures 18 and 19. These tests were performed with grit blast Sol-Gel as well as with grit blast DIARC coated specimens. In the selected reference Sol-Gel process the plates had to be grit blasted no longer than 30 minutes before application of the solution. With DIARC the aluminium surface grit blasting was accomplished in order to get more consistent results. The allowed time span between grit blasting and DIARC coating was 5 days.

All aluminium specimens except bare aluminium with DIARC in fresh water immersion had acceptable initial crack length. The bare aluminium with DIARC in fresh water had initial crack length 46.2 mm which is slightly above the 45 mm acceptance limit used in this study.

In fresh water immersion (Fig. 18) the crack growths of grit blast Sol-Gel treated bare and aluminium specimens were 28 and 18 mm in 48 hours, respectively, being above the acceptance limit 6 mm in 48 hours. In salt water immersion (Fig. 19) the crack growths of grit blast Sol-Gel treated bare and clad aluminium specimens were higher, being 52 and 44 mm in 48 hours, respectively. With DIARC treatment the crack growth was acceptable in both immersions.

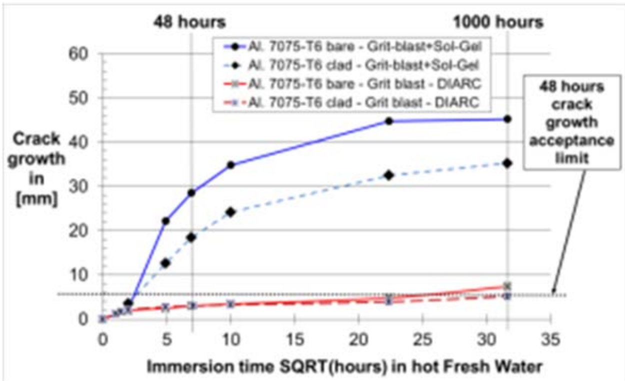

Fig. 18. Crack growth of bare and clad 7075-T6 aluminium wedge test specimens after grit blast Sol-Gel and grit blast DIARC surface treatment methods, hot (60 °C) fresh water immersion.

The failure modes of Sol-Gel specimens in both hot water immersions were all adhesive (Fig. 20). The failure mode of bare aluminium with the DIARC treatment was in both cases acceptable, 90 % cohesive. However, with clad aluminium DIARC specimens the failure mode was in both baths unacceptable, only 30 % cohesive.

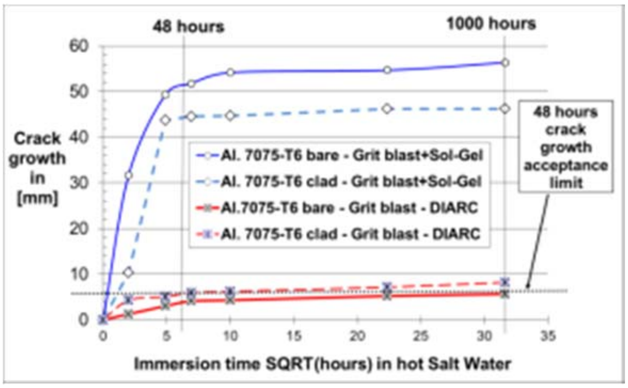

Fig. 19. Crack growth of bare and clad 7075-T6 aluminium wedge test specimens after grit blast Sol-Gel and grit blast DIARC surface treatment methods, hot (60 °C) salt water immersion.

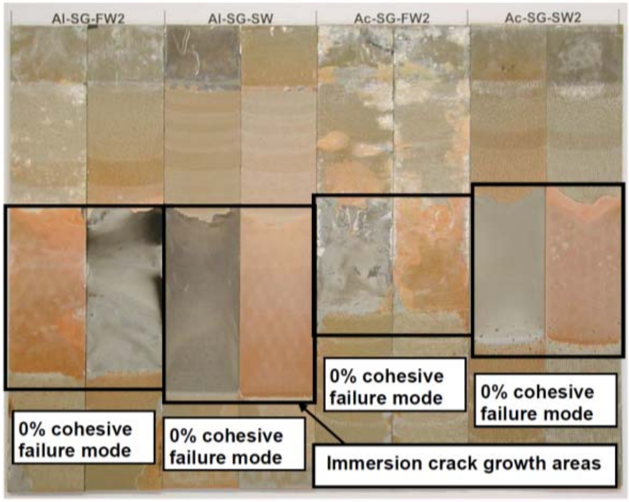

Fig. 20. Typical failure modes of bare and clad 7075-T6 aluminium specimens (Al and Ac respectively) prepared with grit blast Sol-Gel (SG) treatment after 1000 hours immersion at hot (60 °C) fresh (FW) and salt water (SW).

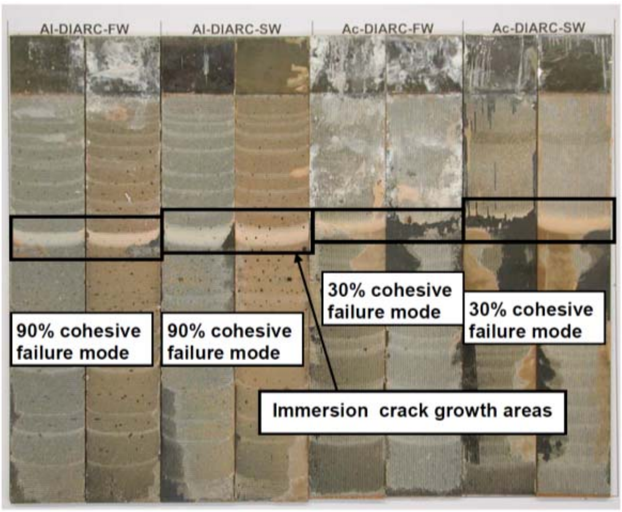

Fig. 21. Typical failure modes of bare and clad 7075-T6 aluminium specimens (Al and Ac respectively) prepared with grit blast DIARC treatment after 1000 hours immersion at hot (60 °C) fresh (FW) and salt water (SW).

4.5 Wedge testing in salt fog

DIARC processes were also tested in a neutral salt fog chamber at 35 °C. The best surface treatment methods found in previous testing [3] were selected for these tests. It means that aluminium plates and high strength steel specimens were grit blasted no longer than 5 days before the DIARC coating. The high strength steel specimens were individually cut, grit blasted and DIARC coated from all edges. The average crack growth values measured in salt fog wedge tests of steel, titanium and aluminium specimens are shown in Fig. 22 - 24.

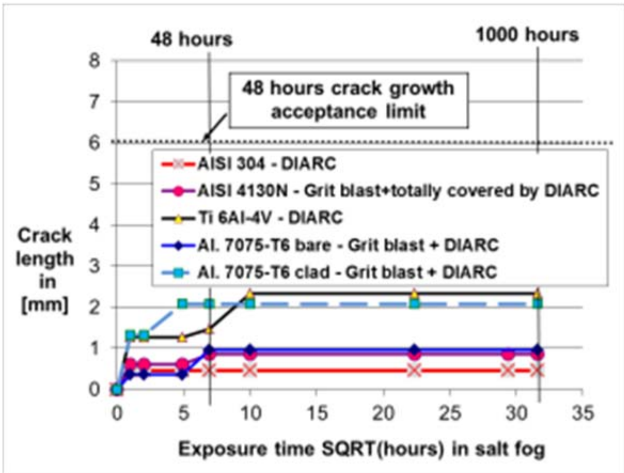

Fig. 22. Crack growth of stainless steel AISI 304 (SS), high strength steel AISI 4130N (SH), titanium and bare and clad 7075-T6 aluminium specimens after DIARC surface treatment at 35 °C neutral salt fog.

Salt fog specimens had the average initial crack lengths 30.3, 36.7, 40.3, 45.8 and 37.5 mm for stainless steel, high strength steel, titanium, bare aluminium and clad aluminium, respectively. The crack growth rates in all specimens were very low. In 1000 hours the crack growths in all specimens were below 2.4 mm.

There was, however, much more variation in the failure modes. Titanium specimens had 100 % cohesive failure mode (Fig. 24). Bare and clad aluminium specimens had 38 % and 50 % cohesive failure modes, respectively (Fig. 23). The failure mode of stainless steel specimens was only 10 % cohesive (Fig. 24).

The failure mode of high strength steel bondline was 56 % cohesive. The corrosion did, in the other hand, penetrate through the thin DIARC coating from all outer surfaces of the specimens and thus caused excessive corrosion in the parent material.

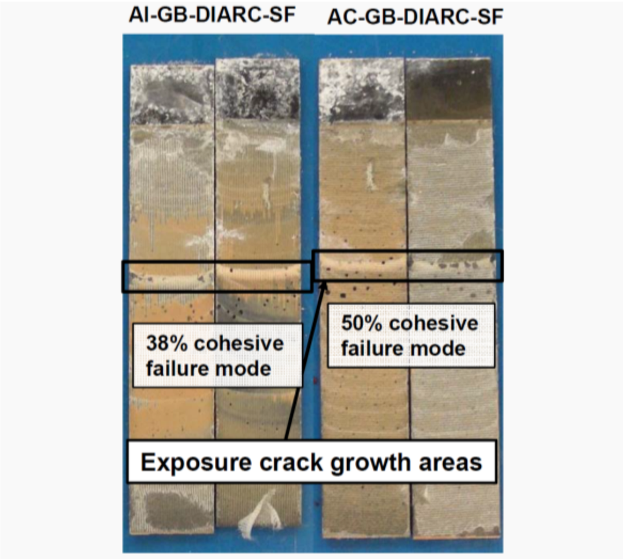

Fig. 23. Typical failure modes of bare (Al) and clad (AC) 7075-T6 aluminiums prepared with DIARC treatments after 1000 hours exposure at 35 °C neutral salt fog.

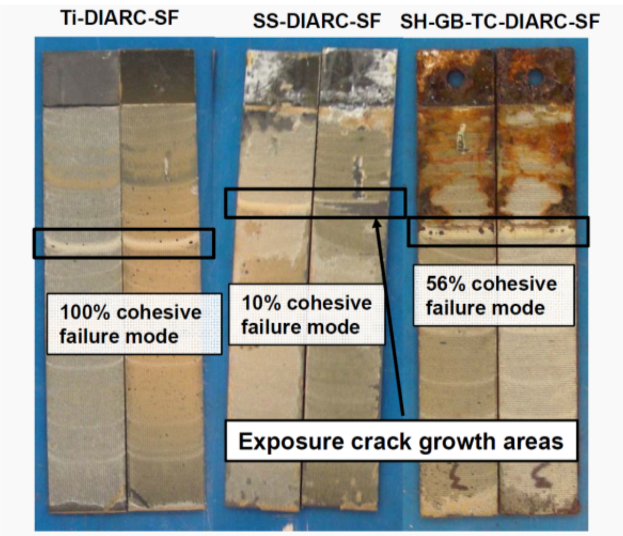

Fig. 24. Typical failure modes of titanium, stainless steel (SS) AISI 304 and high strength steel (SH) AISI 4130N prepared with DIARC treatments after 1000 hours exposure at 35 °C neutral salt fog.

4.6 Case study: Steel insert testing

4.6.1 Adhesion comparison with single lap shear specimens

The adhesion of the insert potting compound EA9396 to stainless steel was screen tested with three methods: no surface treatment (current procedure), grit blast Sol-Gel with primer and DIARC. Single lap shear specimens were used. The tests were accomplished at room temperature as dry and wet. Each set had two specimens.

Average shear strength values (force divided by joint area) measured in the surface treatment comparison are shown in Fig. 25. The performance showed similar trend between the treatments than found with the FM300-2 film adhesive (see Fig. 3). Without any surface treatment the specimens had low shear strengths and mostly adhesive failure modes as dry and as wet. Typical adhesive failure mode is shown in Fig. 26. The grit blast Sol-Gel primer and DIARC treatments performed well and resulted in cohesive failure modes.

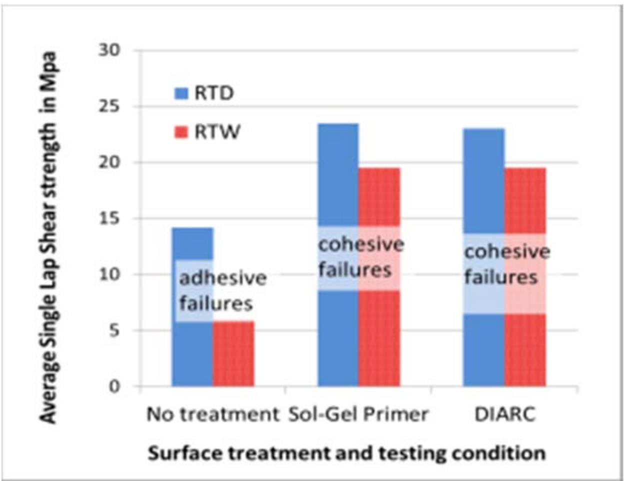

Fig. 25. Measured average single lap shear strength of stainless steel AISI 304 specimens with no treatment, grit blast Sol-Gel primer treatment and DIARC treatment, bonded with EA9396 potting compound and tested at room temperate as dry (RTD) and as wet (RTW).

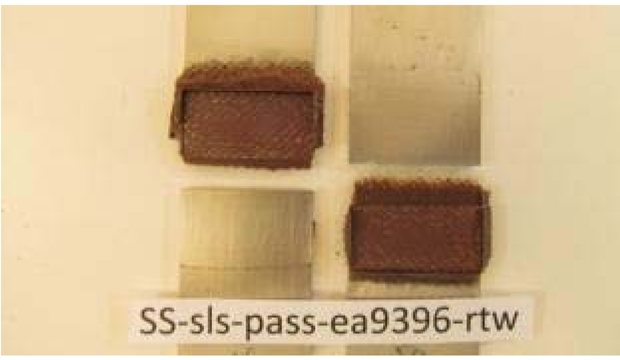

Fig. 26. Typical adhesive failure mode of stainless steel AISI 304 (SS) single lap shear specimens bonded without treatment with EA9396 potting compound and tested at room temperate as wet (RTW).

4.6.2 Insert installation torque testing

The passivated stainless steel 1.4301 DIN 17440 (~AISI 304) insert torque tests were performed with unexposed and exposed specimens at room temperature [6]. The specimens are divided into two groups since the height of the sandwich panels was different. Three inserts were tested in each set.

Uncoated inserts failed from the interface between the insert and the resin compound approximately with 10 Nm torque load as shown in Fig. 28. Failure mode was adhesive with all specimens (Figure 27a).

Both pristine and replaced insert installations were tested in the study. Small difference in strength was found between the uncoated pristine and replaced inserts (see Fig. 28). This difference can be a consequence of a varying degree of filling with the potting compound.

Unexposed coated inserts did not rotate but the bolts themselves failed in torque shear. The bond line between the insert and potting compound was visually found to be intact (Figure 27b). The highest torque moment was 27.47 Nm (Fig. 28).

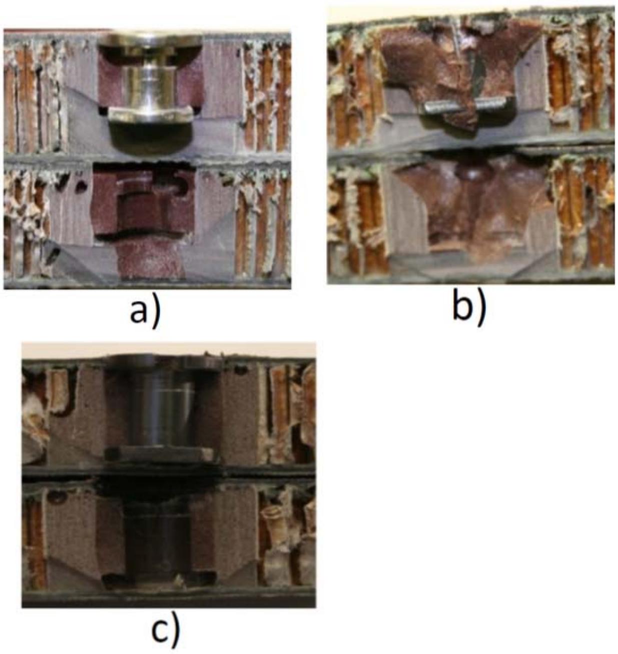

Fig. 27. Failure modes: a) adhesive for the uncoated insert. b) Cohesive for the unexposed coated insert. c) Adhesive for the exposed coated insert.

Coated specimens were exposed 56 days in a typical accelerated hot wet aging environment 60 °C / 98 % RH. Some of the exposed coated inserts rotated, but typically an upper flange of the insert cracked before rotating. The highest torque moment when the cracked insert rotated was 23.70 Nm (Fig. 28). The failure mode was mostly adhesive (Figure 27c).

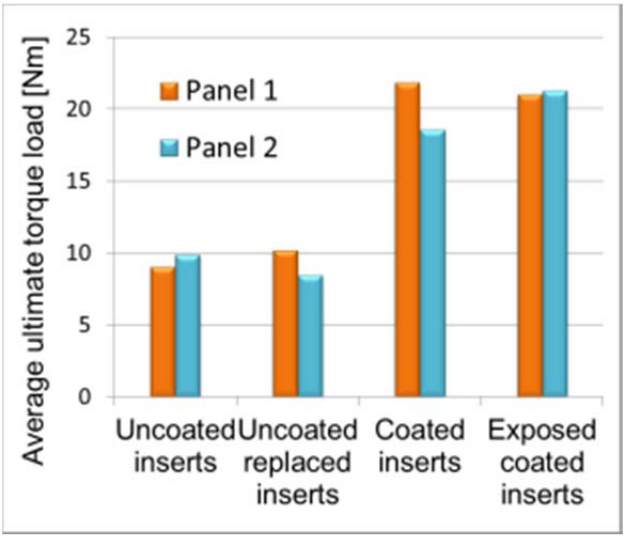

Fig. 28. Measured average ultimate torque loads for uncoated and DIARC coated inserts.

5 Discussion

5.1 Steel bonding

5.1.1 Durability of stainless steel bonding

The stainless steel static single lap and double lap shear testing showed that good performance can be achieved with the DIARC coating. The failure modes were in all tests 100 % cohesive in short single lap shear joints as well as in longer double lap shear joints. In stainless steel single lap shear tests also the grit blast Sol-Gel primer showed good performance.

In wedge testing DIARC coated stainless steel specimens provided acceptable results while grit blast Sol-Gel provided unacceptable results. In hot fresh and salt water immersion testing only DIARC coating was used. In fresh water immersion all values were acceptable. In salt water immersion and in salt fog chamber crack growth was low but the failure mode was unacceptable.

To summarize, the DIARC treatment increased the durability of the bonded steel joints to the level that is typically required for aircraft applications. The stainless steel DIARC wedge test results were comparable to the results achieved in aluminium and titanium wedge testing with grit blast silane and grit blast Sol-Gel primer methods.

5.1.2 Durability of high strength steel bonding

AISI 4130N is a more problematic material than stainless steel since it is very corrodible. The DIARC surface treatment process evolved during the study. The existing corrosion stains on the surface must be removed with grit blasting before the coating and all edges must be coated with DIARC.

Individually bonded specimens were wedge tested in hot salt water immersion and in salt fog. In 0.5 % salt water immersion the results were as good as with the other DIARC coated specimens. The failure mode was cohesive.

In salt fog chamber the results were conflicting. The bondline was the best protected area against corrosion. The crack growth between interfaces was slow and some degree of cohesive failure was observed after opening the specimens. However, the corrosion penetrated through the thin coating and the coupons had heavy corrosion on all other surfaces.

5.2 Titanium bonding

The titanium static single lap and double lap shear testing showed good performance with the DIARC coating. The failure modes were either 100 % cohesive failure in the bondline or titanium adherend failure.

DIARC treatment was referenced in hot wet wedge tests against the qualified grit blast AC-130 Sol-Gel primer method. Both methods provided acceptable performance.

Further tests in hot fresh water and hot salt water immersions proved the good durability of DIARC with titanium. DIARC worked well also in salt fog chamber providing very low crack growth and 100 % cohesive failure mode.

5.3 Aluminium bonding

The DIARC treated aluminium single lap shear specimens showed sufficient performance since failure modes were cohesive and average shear strength values were above or close to typical acceptance limit values. In most DIARC coated aluminium double laps shear specimens the full capability of the adhesive joints was reached and the failure occurred in the adherend.

In hot/wet wedge testing durable aluminium bonding was achieved for bare 7075-T6 aluminium with the grit blast silane method without primer and with the DIARC treatment. For clad aluminium good results were achieved without clad removal and without a primer using Sol-Gel method. The DIARC method without clad removal also provided acceptable results.

In neutral hot water and in salt hot water immersion the grit blast AC-130 Sol-Gel method did not provide any acceptable results with 7075-T6 bare and clad aluminiums. The DIARC coating provided good results also in hot water immersions. In the salt fog wedge testing of DIARC treated aluminiums crack growth was slow, but failure modes were unacceptable.

5.3 Case study: strength and durability of bonded inserts

The installations of DIARC coated steel inserts were in this study always stronger than the ultimate torque shear strength of the steel M5 12.9 bolt. After exposure the coated inserts still had nearly 200 % improved torque strength when compared to uncoated inserts.

6 Conclusions

According to this study, the DIARC treatment appears to be a promising method for durable bonding of stainless steel, titanium and aluminium.

Advantages of the DIARC method for stainless steel and titanium are that no grit blasting is required. For aluminium grit blast is recommended no longer than five days prior to bonding. For all tested metals there is no need for hazardous materials when the DIARC treatment is used. The quality of treatment stays constant and is not affected by the workman skill levels. DIARC treatment can become cheaper since multiple working hours can be saved.

On the other hand, there is a limitation that currently the coating can be done only in a separate vacuum chamber. Also, if used with high strength steel in a highly corrosive environment, the DIARC treatment may need more testing with additional corrosion protection on the surfaces outside the bondline.

The results presented here may be sufficient for some applications for starting to use the DIARC treatment for structural bonding. However, in future more tests are needed to deteremine, for example, the fatigue behavior of bonded joints with the DIARC coating.

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