Critical Steps in Adhesive Bonding Process for Space Applications

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Outline

- Adhesives in space applications
- Challenges for adhesive bonding
- ESA’s activities on adhesive bonding for space applications
  - ECSS-Q-ST-70-16C, status
  - Activities to support MPTB (selected adhesives with recent change in formulation)
- Parameters affecting durability of adhesive joint
  - Surface treatments: Invar vs. aluminium alloy
  - Curing temperature: RT vs. high temperature post-cure
- Training and certification in adhesive bonding (EAB, EAS, EAE) schemes
- Factor of bonding operator on quality of the joints
Adhesive bonding – Process nr. 1

- Synthetic industrial adhesives are being produced for decades and are massively used by space industry (light weight structural joint, stress distribution, dissimilar adherents, low cost)

- Variety of use:
  from very special cases as bonding (cover glasses on solar cells), optical element bonding (lens and mirror bonding), up to robust joints in structural applications (honeycomb core to CFRP laminates)

- Adhesives classifications:
  - chemical nature/cure (Thermosets, thermoplastic, elastomeric)
  - function (structural, non-structural, special or combined functions – conductors, insulators, gap-fillers, tapes, sealants, thermo-optical functionality)
  - application (1-C, 2-C, foil adhesives, PSAs)
Challenge for materials and associated processes

Space missions are demanding the highest possible performance of used materials.

Examples of extreme ESA mission’s requirements:
BepiColombo (thermal endurance, (V)UV & particle radiation, extreme temperature range given by mission profile, cycling)

-190; +450 °C

...validation of materials and verification of processes can become more challenging than the mission itself -> necessity to push the boundaries of available technologies to achieve scientific goals of the missions.
Why adhesives fail in space applications?

From lessons learned...

- **Bulk properties under/over estimated** (wrong material selection -> impact on design)
- **Venting not considered** in design (weakening of adhesive + outgassing pressure build-up at elevated temperature = bubbling)
- **Curing parameters not heeded** (pot life, set time, time in fixture) => misalignment of optical instruments, mechanical detachments
- **Tg crossing conditions** + applied loads => creep or too brittle (impact on assembly)
- Rapid **thermal decomposition** => explosions in thermal vacuum (bubbles, cracks through thin adherents)
- **Insufficient resistance to accelerated testing** conditions (synergy of T, RH, TvaC... UV/VUV, p+, e-, chemical instability at the interface)
- Long term **operation outside service temperature range** (often subject of qualification plans, TDS allows “short” periods but what does that mean for Tvac applications?)
- **Secondary function failure** with impact (not considered when accepting primary function; e.g. optical, discoloration, contamination of other components)
Impact of etching on optical glass bonding

- Etching of **optical glass ceramics** helps to remove micro cracks but brings another challenge (mastering of etching, neutralization and cleaning processes)
- Simple UV-illumination within incoming inspection to detect the issue
- Impact on bonding performance can be significant

Inconsistent layers of surface treatment residues can endanger adhesive bonding

-> need for further investigations
Impact of etching on optical glass bonding cont.

- Chemical analysis (SEM-EDX, XRD) to identify the residue composition
- Understanding of composition can help in selection of correct techniques for removal (might be challenging)

Example of bulk composition of lithium-alumino-silicate glass-ceramic:

\[ \text{SiO}_2, \text{Al}_2\text{O}_3, \text{P}_2\text{O}_5, \text{Li}_2\text{O}, \text{TiO}_2, \text{ZrO}_2, \text{ZnO}, \text{MgO}, \text{As}_2\text{O}_3, \text{K}_2\text{O}, \text{Na}_2\text{O} \]

the content of the main elements is carefully tuned to achieve the “zero” CTE glass

after etching:
- “conversion layer interface” composition different
- thermo-mechanical properties and adhesion are likely to be different
- surface morphology is strongly dependent on concentration of acid, immersion time and rinsing methods

Elemental composition of “white” residue

K, Ca, Na, Ti, Mg, Al, Zr, Zn, O, C, F, Si, Zn
ECSS-Q-ST-70-16C under public review

Adhesive bonding for spacecraft and launcher applications

What do we expect from the standard?

- applicable to all European space programmes (PA requirements)
- provide a baseline for R&D projects on new space adhesive development (air-vacuum shift, thermal stability, adhesion to substrates)
- give cheap and quick screening tool to assess various types of adhesives and adherents (similar to outgassing in contamination screening)
- help to identify unusable substrate-adhesive combinations, can also invalidate the particular adhesive or disqualify particular adhesive bonding process

- helpful tool for the space industry & SMEs to verify their bonding process and to identify prospective adhesive/primer/substrate combinations for space applications

WG group members:
IFAM Fraunhofer Institute, RUAG Space, Thales Alenia Space, Airbus Defence & Space & Safran Launchers (Ariane Group), ESA, CNES, Leonardo Company (Finmeccanica), OHB Systems, ESA/ECSS secretariat
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Adhesive bonding for spacecraft

What do we expect from the standard?

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Assessment of chemical formulation change aerospace grade 2-C Epoxy

Objective: Quantification of the impact of formulation change by comparing the initial formulation with the modified version (M).

<table>
<thead>
<tr>
<th>Step</th>
<th>Test</th>
<th>Sub-objective</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>ASTM D1002, Lap shear tests</td>
<td>Determination of shear properties of adhesively bonded single lap joints</td>
<td>+2%, negligible, ok</td>
</tr>
<tr>
<td>2</td>
<td>Lap shear tests according to DIN EN 14869-2</td>
<td>Determination of shear modulus of adhesively bonded single lap joints (thick adherents)</td>
<td>Shear modulus: -50%</td>
</tr>
<tr>
<td>3</td>
<td>DCB test according to ISO 25217</td>
<td>Determination of adhesive fracture energy $G_{IC}$</td>
<td>$G_{IC}$: -8%, ok</td>
</tr>
<tr>
<td>4</td>
<td>Boeing wedge tests according to ASTM D3762</td>
<td>Determination of the environmental durability in water at room temperature</td>
<td>CGR same, pattern, ok</td>
</tr>
</tbody>
</table>

in cooperation with IFAM Fraunhofer, 2012
Properties of RT-cured EC2216 and EC9323-2, “new” vs. “old” formulations

Preliminary results ISO 527-2 (RT cured, new vs. old versions)

According to first preliminary results no dramatic change observed on none of these two modified 3M’s epoxies. Higher impact is noticeable on EC9323-2, with change in selected material properties within 13%. Nevertheless complete set of results is expected by end of 2018

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Modulus (MPa)</th>
<th>Poisson ratio</th>
<th>Max. strength (MPa)</th>
<th>Strength at break (MPa)</th>
<th>Strain at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EC2216 OLD</td>
<td>118 (6)</td>
<td>0.408 (0.021)</td>
<td>17.2 (0.3)</td>
<td>17.1 (0.4)</td>
<td>41.1 (1.8)</td>
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<tr>
<td>EC2216 NEW</td>
<td>133 (10)</td>
<td>0.420 (0.015)</td>
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<td>EC9323-2 OLD</td>
<td>1281 (50)</td>
<td>0.390 (0.022)</td>
<td>27.2 (0.7)</td>
<td>27.0 (0.7)</td>
<td>3.01 (0.56)</td>
</tr>
<tr>
<td>EC9323-2 NEW</td>
<td>1158 (101)</td>
<td>0.398 (0.016)</td>
<td>24.8 (0.5)</td>
<td>24.8 (0.6)</td>
<td>3.24 (0.43)</td>
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Relative change

in collaboration with Rescoll

According to first preliminary results no dramatic change observed on none of these two modified 3M’s epoxies. Higher impact is noticeable on EC9323-2, with change in selected material properties within 13%. Nevertheless complete set of results is expected by end of 2018.
Surface treatments and post-cure: key steps towards joint endurance after hot/wet exposure

Study dedicated to surface treatments efficiency of **FeNi36 alloy (INVAR®)**, with **ScotchWeld2216** (old formulation), single lap shear, EN1465, 2days RT cure, **2H 65degC post-cure**; hot/wet conditions: 7days, 95%RH, 55°C

**motivation:** frequent failures of bonding after hot-wet exposures (results often scattered, low adhesion strength)

**results:**
- clear improvement in strength for both conditions (**+40%**)
- shift from (adhesive/ interfacial failure mode to cohesive fracture mode
- regardless the surface treatment, adhesive is more resistant against moisture with high-temperature post-cure step

<table>
<thead>
<tr>
<th>Various surface treatments</th>
<th>Single lap shear strength (MPa)</th>
</tr>
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<tbody>
<tr>
<td>Mechanical</td>
<td>RT: 12.75, after hot/wet: 13.48</td>
</tr>
<tr>
<td>Mechanical -&gt; Degrease</td>
<td>RT: 18.04, after hot/wet: 17.67</td>
</tr>
<tr>
<td>Mech. -&gt; Degrease -&gt; Primer</td>
<td>RT: 21.64, after hot/wet: 20.66</td>
</tr>
<tr>
<td>TDS value (Etched Aluminium)*</td>
<td>22.06, 20.33</td>
</tr>
</tbody>
</table>

* TDS hot-wet conditions: 14days, 100%RH, 49°C, Aluminium, TDS data 2009

in collaboration with Rescoll
Enhanced sensitivity to moisture with RT cure

Study dedicated to surface treatments efficiency of FeNi36 alloy (INVAR®), with ScotchWeld2216 (old formulation), single lap shear, EN1465, 7 days RT cure, no post-cure; hot/wet conditions: 7 days, 95%RH, 55°C

motivation: to simulate frequent failures of bonding after hot-wet exposures reported on INVAR

results:
- Surface treatment improved mainly strength of references
- clear susceptibility of adhesion strength to moisture (in some cases -55%)
- regardless selected surface treatment, RT-cured joints were relatively more susceptible to moisture when compared with post-cure (previous slide)

in collaboration with Rescoll
Enhanced sensitivity to moisture with RT cure

Study dedicated to surface treatments efficiency on aluminium alloy 2024 T351, with ScotchWeld 2216 (old formulation), single lap shear, EN1465, 7 days RT cure, no post-cure; hot/wet conditions: 7 days, 95%RH, 55°C

motivation: to compare with results from INVAR

results:
- Surface treatment improved mainly strength of unexposed joints (similar to INVAR)
- clear susceptibility of adhesion strength to moisture (in some cases -40%)
- improvement in fracture mode, but not so efficient as HT post-cured
- regardless the surface treatment, adhesive joint is susceptible to degradation when exposed to hot-wet conditions (similar to INVAR)

in collaboration with Rescoll
European Adhesive Bonding training scheme

- adopting the philosophy of EWF training for welders

**Professional Qualification**

- Combined System EWF and IAB/IW ANBs
- Rules for the Approval of ANBs and ATBs
- EWF and IAB/IW

**EWF System**

**Qualification Levels /Guidelines:**
- IWE/EWE - Engineer
- IWT/EWT - Technologist
- IW/EWS - Specialist
- IWIP/EWI - Practitioner
- IW/E - Welder (3 levels, 4 processes)
- IWSD - Structures Designer (2 levels)
- Distance Learning

**Guidelines:**
- EAE/EAS/EAB - Adhesive Bonding
  - EWS/EWT/EWIP/EW/WR - Resistance
  - ETS/EYB/ETS - Spinning
  - MMA EW-Diver
  - RPM - Plastic Welder
  - European Aluminothermic Welder - EAW, RAILSAFE/Guide/PJ/50/02/00227

**Special Courses at Specialist Level:**
- Laser
- Robots
- Reinforcing Bars
- Weld Imperfections for NDT Pers.
- Macro and Micro Examination
- Heat Treatments
- Risk Management in Welding Fabrication

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**The international Training and Qualification system for Personnel**

<table>
<thead>
<tr>
<th>Route 1</th>
<th>Route 2</th>
<th>Route 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>EAB (bonder)</td>
<td>EAS (specialist)</td>
<td>EAE (engineer)</td>
</tr>
<tr>
<td>40h+EAB exam</td>
<td>40h+EAB exam</td>
<td>332h+EAE exam</td>
</tr>
<tr>
<td>0+120h+EAS exam</td>
<td>EAB+80h+EAS exam</td>
<td></td>
</tr>
</tbody>
</table>

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Operator’s influence on slap shear strength (EN1465)

motivation: to compare results within group of operators, each of them to prepare own samples, surfaces, mixing the adhesive, application, curing and testing. Further comparison with external lab – reference values

results:
- each operator achieved slightly different results (absolute strength vs. reproducibility)
- portion of adhesive failure correlated with strength of each joint (varies, operator to operator, details on next slide)
- for structural applications the operator with lower average strength but with better reproducibility could reach better design allowable (A or B values or even 3xsigma)
Conclusions

- adhesive manufacturers change formulations and processing procedures regularly! -> need to check the quality within incoming inspections (bulk property)
- for EC2216 and EC9323-2 assessment of formulation changes ongoing
  - (from Modulus, Poisson ratio, Strength and Elongation at break no big difference)
- ECSS-Q-ST-70-16C is under review, over 100 unique requirements and countless recommendations to achieve proper adhesive bonding
- for selected 2-C epoxies the post-cure at elevated temperatures leads to better durability of the joints when exposed to “hot-wet” conditions when compared with RT-cure equivalents
- thoroughness of surface treatments affects the shear strength greatly, peel strength is expected to be even more affected (initiation of delamination)
- even with same process parameters the “operator’s effects” should not be underestimated
“....Quality is not a result of testing, it must be an integral part of the manufacturing system!” [5]  

Prof. Dr. rer. nat. Gerd Habenicht

Thank you for your attention
Questions?

**Paper 99**: Critical Steps in Adhesive Bonding Process for Space Applications

**Poster & Paper 100**: Surface Analysis of Solar Cell Cover Glasses and OSRs after High Temperature UV and VUV Exposure