CertBond
COST Action CA18120

WG3: Manufacturing phase

- Guidelines for the optimum manufacturing processes
- Guidelines for testing methods during the manufacturing process

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Preamble - Introduction

Bonding may seems to be a really easy process to join parts. Everyone has done it at home with successful examples without neither reading the user's notice of the glue nor using complex procedures!

However, in an industrial environment, reliability of the bonded joints is mandatory and the use of "artisanal" processes will lead, at more or less long-term, to unexpected and potentially critical failures.

To reduce this risk, strict procedures must be implemented and followed for the all the phases on an industrial bonding process. A lot of books, handbooks, formations can be used to help the person in charge to establish these procedures and to help the operators to work "well".

This document has not the vocation to replace all these tools but rather to give useful "tips" based on the know-how of several industrial manufacturers of bonded parts. These tips were received during interviews with specialists from various aerospace companies. A summary of these interviews can be found at the end of this document.

In the original CERTBOND program, two different deliverables were planned to cover both the manufacturing process and associated tests. On the other hand, in practice, these two domains are fully intricate and, therefore, this document bundles the two domains.

Guidelines for manufacturing of bonded joints

- Before the bonding
  - Optimize the design of the bonded joint: follow the “good practice” rules when designing the assembly. These rules take into account the specificities of adhesive when compared to mechanical fasteners and can be found in many handbooks, manuals, etc. Amongst those rules, the three ones describe below are a good start :
    - maximize the bonded surface (and therefore reduce the strain within the joint)
    - reduce stress concentration and singularities
    - avoid peeling zone as adhesives are intrinsically weak for this type of solicitation
  - Know the environmental conditions that will be applied to the joint: the mechanical loadings is not the only factor that should be taken into account but to assure a right ageing of the assembly, environmental conditions play a key role : temperature (max but also min), fluids, etc.
  - Selection of the adhesive: try to use quantitative requirements
    - In many case, when an adhesive is already use in the company, the first option of the design team is to stay with this known solution event if the requirements are different. And when an adhesive change is needed, one has often the specification from the manufacturer that the new adhesive “should be better” than the actual one.
    - The adhesive properties result from a delicate balance between contradictories effects and the objective to improve everything at the same time is quite an impossible task.
    - On the contrary, if you have a quantitative evaluation of the adhesive solicitations (e.g. for the mechanical performances, based on calculus made during the design phase), you can base your specifications/requirements on the good parameters and optimize your selection
    - Once you have this requirements, don’t trust to much the manufacturer’s data you can find e.g. in technical data sheets: they are given for information only, they may have been determined in systems different than your application and negative points could have been hidden! These data should only be the first selection filter, before doing your own experimental plan, based on your specific needs.
  - Always have a qualification phase, adapted to the criticity of your parts: the more the bonding is critical for the structure, the more you should experimentally test your solution. Below are some of the requirements for aerospace assemblies:
    - Test the adhesion on you specific substrates, and with exactly the same surface preparation

        - Don't forget to test the ageing behaviour of the assembly. In many cases, hot wet ageing (with ageing conditions that depend on the industrial domain) covers the other solicitations but there are exceptions
        - Validate the curing cycle, and in particular for aerospace, verify that the adhesive is fully cured (as close as 100% as possible) for a better stability of the adhesive properties with time.
        - Check the limit of the process to control your process margins: open time, mixing ratio for 2K, etc.
        - At the end of the qualification phase, fix the process parameters and prepare an adapted documentation
    - Train the operators
        - Many bonding trainings, with various level of technicity are available. The operators do not need to be expert but they should at least know the basics, in order to understand the reason the bonding procedures must be "strict"
        - Without such knowledge, you will have sooner or later deviations with unpredictable results

- During the bonding process
    - Surface preparation is mandatory, as it allows to bond on a known surface state
        - Degreasing/sanding/degreasing is already a good option, that works for a significant amount of materials, especially when the environmental conditions are "soft"
        - In more critical cases, more complex (and specialized) process may be necessary
    - Be careful with the environmental conditions (Temperature, Relative Humidity) : you don't always need a clean room nor an air-conditioned space, but most the adhesive have limitations concerning the ambient temperature and relative humidity
    - Follow strictly the procedures made during the qualification phase
    - Register as many parameters as you can, while maintaining the good balance between the criticity of the bonding and the extra work needed for the records.
    - In case of you detect any problem, stop and analyse! Don't expect that the problem will resolve itself. On the contrary, use the recorded data, try to understand what happened, propose a solution and test it before returning to the serial
    - If you have large amount of bonding, automatisation (surface preparation, adhesive deposition, parts accosting) is a good way to reduce variability and therefore the risk.

- After the bonding process
    - If you joint is cured under temperature, check systematically your temperature record to check if you have followed the validated cure cycle

- There is at present no validated method to measure the final mechanical performance even if it is an active research domain with promising results. However, for critical bondings, classical Non Destructive Technique methods, such as the most commonly used Ultrasonic, may verify the homogeneity of the joint (lack of adhesive, bubbles, and porosities). You may also do destructive mechanical testing on additional samples, even if this could not be consider as a full proof of quality.
- Stay aware of long term problems, like disbonding after several months or years of use. It may be due to solicitations (mechanical/environmental) higher than expected but also could be a signal of a design/manufacturing problem. Therefore, all these returns of information should be investigated. . In such case, having good archives of the process data is crucial for understand the problem and eventually propose corrective methods for future applications. To adapt the effort due the archive's conservations, the conservation's duration should be adapted to expected lifetime of the bonded parts.

Extra reading

- Adhesive Bonding Science, Technology and Applications,2nd Edition - June 30, 2021,Editor: Robert D. Adams Paperback ISBN: 9780128199541,eBook ISBN: 9780323851435
- Advances in Structural Adhesive Bonding,2nd Edition - June 10, 2023,Editor: David A. Dillard,Paperback ISBN: 9780323912143, eBook ISBN: 9780323984379
- Handbook of Adhesion Technology - 07 June 2018, EditorsLucas F. M. da Silva, Andreas Öchsner, Robert D. Adams, ISBN : 978-3-319-55410-5
  Adhesive Bonding in Five Steps: Achieving Safe and High-Quality Bonds, 4 February 2022, ISBN:9783527349142

Examples of training

- https://www.weiterbildung.ifam.fraunhofer.de/en/adhesive-bonding-technology.html
- https://www.twitraining.com/home/programmes-and-courses/other-specialised-engineering-courses/adhesive-bonding-technical-workshop
- http://www.inegi.pt/en/programs/advanced-joining-processes-course-adhesive-bonding/
- https://www.skz.de/en/training/courses/adhesive-bonding
- https://www.kth.se/student/kurser/kurs/MG2037?l=en
- https://www.delo-adhesives.com/us/academy/seminars
- https://rescoll.fr/formations/
- https://www.cetim.fr/formation/formation/production/Procedes-d-assemblage/Collage
- https://www.udemy.com/course/basics-of-adhesive/
- https://learn.toolingu.com/classes/intro-to-adhesive-bonding-110/

- https://www.kiwa.com/tr/en/service/training/adhesive-bonding-training-eab-eas/

Summary of the industrial survey

A survey has been done by the WG3 members with industrial partners, selected within the aerospace domain. These interviews were based on a list of questions, covering most of the aspect of bonding.

The results are presented below (excluding the confidential information's) and show common practices, but also some divergences due to the differences in the use of the bonded parts.

Here are presented the main results from this survey with highlights on common practices.

- **Requirements/Specifications**
  - In most cases, they are aligned with that of the suppliers
  - If some additional constraints are needed, they are generally included in a so-called PCD (Process Control Data) that specify specific rules on the ingredients, the process, the number and localization of the qualified plan, the obligation on documentation These PCD is negotiated via the commercial contract.
  - For aeronautic products, specific aeronautic grades (with more quality controls) are always uses, at least for structural bonding
- **Batch reception and acceptation**
  - In most cases, done based on the conformity certificate issued by the supplier, without additional in-house control
- **Shelf-life (at storage temperature and at ambient)**
  - This time limitations are taken into account with procedures.
  - For each batches (or mix for 2K adhesives), all the data are recorded and can be linked to each bonded parts
  - These limitations can be more drastic than the ones given by the supplier (e.g. for 2K paste, if the rheology evolution of the paste is detrimental to the application)
  - For 1K adhesive, procedures are available to extend the storage time. Typically, by making physical-chemistry analysis, it is possible to extend the storage time by 50% of the initial duration.
- **Surface preparation**
  - Everybody impose a proper surface preparation before bonding, at minima a degreasing
  - This surface preparation is always consider as a "special process" (in the sense of the aeronautical regulations)
  - For metals, we have in general a chemical etching and/or conversion followed by the deposition of a primer. The role of the primer is in general double: corrosion protection and extension of the open time before bonding (up to 6

months). For less critical bonding, one could find also sandblasting (or grinding) +degreasing preparation, with open time limited to a few hours.
    - For CRFP, the preferred surface preparations is sandblasting (or grinding) +degreasing. One can also uses specific peel-ply that are removed just before the bonding.
    - For elastomer, a chemical adherisation (after sanding/grinding) is generally needed for a good adaption between the chemistries of the adhesive and the elastomer.
    - Many industrials are interested by automatized processes, like laser or plasma.
    - The process parameters are selected and fixed during the qualification phase. The performance of the surface preparation is in general measured by mechanical testing on standard specimen (with ageing). Measurement of the surface energy or of the rugosity is sometimes used, but it is often difficult to have good correlation between these measurements and the mechanical performances.
    - For the production, the process parameters are recorded and attached to the documentation of the final part.
    - In production, there is in general no control of the surface quality after the preparation. In some specific cases, mostly old programs, one can do water break test (for an estimation of the surface energy) or periodic destructive mechanical testing on accompanying samples.
- **Bonding**
    - The key parameters of the process are evaluated during the qualification phase, then fixed in the documentation and recorded during production.
    - The ambient conditions for the bonding are very variable, from clean room (no dust, Temperature and Relative Humidity RH stabilized) to standard workshop. But at least, the temperature and RH should be recorded and limitations should be determined, depending of the properties of the adhesive.
    - The curing cycle may be different from that proposed by the supplier, in order to adapt to specific constraints and/or to improve the reticulation % when compared to the standard cycle (when bonded parts are submitted to long term high temperature, one tries to be as close as 100%).
    - For curing in temperature (oven or autoclave), the cure cycle may be adapted to the number and positions of the parts within the autoclave. The temperature is not recorded within the adhesive joint and the cure cycle is piloted via the “ambient” temperature of the autoclave. All the temperature/pressure records are registered within the part documentation.
    - The gap between the 2 bonded parts (final adhesive thickness) is managed by different ways. For film adhesives, the thickness is adjusted by the pressure during curing, thanks to the support of the film that gives a minimal thickness. For paste adhesive, one can found several methods: joint design with e.g. contact zone, use of calibrated fishing line or beads or control of the deposited volume

- The strategies about destructive mechanical testing (after bonding) are various, depending of the final parts. However, in most case, some additional samples are prepared together with the parts and are available in case of specific expertise would be necessary.
- Although all parties record a lot of process data, no systematic analysis are made in general

- **Testing after bonding**.
  - Systematic NDT is only made in the case of large structural parts. For these parts, ultrasonic (C-scan, A-scan) is the method of choice but additional technics could be used (Thermography, Tap test, X-rays control)
  - Also for these parts, specific procedures are used for defect management with the determination of a list of acceptable defects (positions and sizes) and methods for reparation (e.g. by adhesive injection) or justification (by calculus)
  - Periodic sampling of final bonded parts by destructive methods is avoided if possible.

As a summary, all interviewed entities (that all belong to the aerospace domain) use basically the same global bonding processes with a lot a similarities in the detailed implementations.

The two societies who produce the more critical parts (and also the larger ones) add some more controls and procedure, for a more robust process:

- PCD (Process control data): contract with the supplier that defines precisely the fabrication process of the adhesive (e.g. source of raw products, mixing lines…)
- Systematic control by NDT and procedures for defect management
- FAI (fist article inspection) with destructive testing of the bonding

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Questions	Indus #1	Indus #2	Indus #3
Géneral			
Domain	Aeronautic	Aeronautic	Aeronautic
Type of bonding	Mostly sandwich (Epoxy and BMI) Metal/CFRP	Structural Metal/Metal ; CRFP/Metal ; CRFP/CRFP	Elastomers
Special process ?	Yes	Yes	Yes
R&D topic ?	**How to bond on 3D parts ?**		**Surface preparation** **Adherisation (for elastomer)** **Dismantelling - Unbonding**
Step1 : adhesive			
Auxiliary materials	Fixed in the reception requirements and also in the PCD (process control data) of the supplier for the separator films, the supplier process is verified during the qualification phase- need to inform us if any change	Fixed in the reception requirements and also in the PCD (process control data) of the supplier for the separator films, the supplier process is verified during the qualification phase- need to inform us if any change	No auxiliary materials (paste adhesives)
Requirements > supplier	Yes - based on the qualification phase (specific confiurations) They are shared with the supplier, that should agree within the commercial contract	No	No
Test of each batch	Only those necessary for the producer conformity certificate No internal testing	Only those necessary for the producter conformity certificate No internal testing Except : IRTF spectra comparison and visual inspection	Only those necessary for the producter conformity certificate No internal testing In some particular case : mechanical testing on specific substrates
Time limitation	Yes, managed with several instructions et data	Yes, managed with several instructions et data	Yes, managed with several instructions et data
Step2 : Surface preparation			
Special process ?	Yes	Yes	Yes
Type of surface prep	For CRFP : Peel ply (removed just before bonding) For metals : Sanding/degrasing (open time < 8 h) or chemical preparation (in bath) + Primer (open time : ~ 6 months)	For CRFP : Automated sandblasting (open time ?) For metals : Sanding/degrasing (open time < 8 h) or chemical preparation (in bath) + Primer (open time : ~ 6 months)	manual Sanding/Sandblasting + degreasing + chemical adherisation (primer) if necessary
Following of the process parameters	Fixed during the qualification phase No specific control (could be done by the sub contractor that made the chemical conversion) Periodic control via samples (mechanically tested)	All the process parameters are fixed and followed - everything is recorded in the parts documentation	Rugosity measurement (but operator dependant)

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Questions	Indus #4	Indus #5	Indus #6
Géneral			
Domain	Optical instruments	Aeronautic	Space (civil & militar)
Type of bonding	Glass/metal	Structural/non structural Metals/Plastics/CRFP	Metals/CRFP/Elastomer
Special process ?	Yes	Yes	Yes
R&D topic ?		Not really	Understanding of the adhesion (chemistry) Automatized surface preparations
Step1 : adhesive			
Auxiliary materials	No auxiliary materials (paste adhesives)	No auxiliary materials (paste adhesives)	Auxiliary materials (e.g. separator) are managed using the same process than the adhesiv itself PSA ("scotch"), if they stay on the structure for the flight, are also managed as the adhesives
Requirements > supplier	No Use of standard industrial products (and not aerospace grades)	No,	It may be happend but the tendancy is to try to align with supplier's one
Test of each batch	Only those necessary for the producter conformity certificate No internal testing In some particular case : Xray inspection of cartridge to check for bubbles	Only those necessary for the producer conformity certificate No internal testing	Only those necessary for the producer conformity certificate No internal testing (with some exception) When starting a new serie, additionnal controls may be added (for stability control) temporarely
Time limitation	Yes, managed with several instructions et data	Yes, managed with several instructions et data	Yes, managed with several instructions et data Constraints may be more severe than suplier's data
Step2 : Surface preparation			
Special process ?	Yes	Yes	Yes
Type of surface prep	Several methods, depending on the susbtrate : sandblasting, plasma, laser, direct bonding (glass) If not online, an open time is fixed	In almost all cases, at least degreasing In some cases, sandblasting Spécific cases : atm plasma (gold) No primer, the last opération of the surface preparation is made just before the bonding	Mostly, chemical preparation On metal, bonding on the anti-corrosion complex (e.g. paint) If bonding on primer, degreasing is made juste before bonding
Following of the process parameters	Yes On large series, statiscal analisys will be done	No, manual process in mist cases	All the process parameters are fixed and followed - everything is recorded in the parts documentation

Questions	Indus#7
Général	
Domain	Spatial
Type of bonding	Metals/CRFP
Special process ?	Yes (but with a different definition that in aeronautic : not only visual control)
R&D topic ?	Surface preparation Dismontability SHM of the bonded joint
Step1 : adhesive	
Auxiliary materials	Auxiliary materials (e.g. separator) are managed using the same process than the adhesiv itself
Requirements > supplier	May have supplementary requirements, e.g. degasing or temperature range These additionnal requirements are not known by the supplier and are managed via the control of process and ingredients
Test of each batch	Yes Controls are done by the distributors that repackage the adhesive
Time limitation	Yes, managed with several instructions et data
Step2 : Surface preparation	
Special process ?	Yes
Type of surface prep	Metals : chemical etching + conversion coating (if needed) Composite or non structural bonding : Sanding/degreasing Use of primer for extended open time (but with degreasing)
Following of the process parameters	All the process parameters are fixed and followed - everything is recorded in the parts documentation

Questions	Indus #1	Indus #2	Indus #3
Quality control of the surface prep	No in general (fixed process) For some old programs : water break test or use of control inks	On CRFP : water break test On metals: via mechanical testing of follower samples	No In some case, via mechanical testing of follower samples
Step 3 : Bonding process			
Key parameters ?	A lot of thing are fixed (and listed into the process documentation) Bonding location, cure cycle, parts and adhesive requirements, worker's habilitations....	A lot of thing are fixed (and listed into the process documentation) Bonding location, cure cycle, parts and adhesive requirements, worker's habilitations....	A lot of thing are fixed (and listed into the process documentation) Cure cycle, parts and adhesive requirements, worker's habilitations....
Environnemental condition	Controlled HR and T° Clean room	Controlled HR and T°	HR and T° measured with operation limit
Curing process	Qualification of the curing process For each autoclave, the curing cycle take into account the number and positions of parts Parameters : variation rate (up/down phase) and dwell times Temp is recorded and included in part's documentation The temperature is not recorded within the adhesive joint but in the autoclave (eventually 2 measurements points in Le Havre)	Qualification of the curing process For each autoclave, the curing cycle take into account the number and positions of parts Parameters : variation rate (up/down phase) and dwell times Temp is recorded and included in part's documentation The temperature is not recorded within the adhesive joint(only done during the qualification) but in the autoclave (all the temperature probes are recorded)	Autoclave and under press Curing cycle defined during the qualification phase T° range is generic for several parts The real T° of the part is not recorded during the curing
Gap management	Mostly film adhesive : control via the pressure in the autoclave If the gap is to big, superposition of several adhesive films	Mostly film adhesive : control via the pressure in the autoclave and the tichness of the support tissue) If the gap is to big, superposition of several adhesive films	Not a problem for this type of bonding (contact bonding - very low thickness)
Travelers or cut-off samples	For each curing, travelers specimens are prepared (perpendicular traction, SLS, peeling) but not systematically tested No cut-off samples (especially due to complex part shapes)	No but periodic sampling of parts	No except for some specific applications
Statiscal analalysis	Yes, on process data. Control card may be done for drift identification (but they are very rare)	Everythin is recorded but not systematically analysed	
Step 4 : Quality control			
Systematic NDT	Yes : Thermography - US Cscan or Ascan - Tap test RX (or X tomography) in some cases	US Csan or Ascan	
Management of defects	For each type of parts, a max. deffect size is predetermined (if several deffects, no intercation is mandatory) Defects could be repair (e.g. adhesive injection) or justified by calcul	For each type of parts, a list of acceptable deffects is established (made in the beginning of the production phase) Defects could be justified by calcul and/or after complemtary NDT	
First part qualification ?	Yes (FAI : fisrt article inspection) with destructive mechanical testing	Yes (FAI : fisrt article inspection) with destructive mechanical testing	
Periodic sampling with destructive test	No (except special demand of the client) - Expensive !!	No except for a specific program	

Questions	Indus #4	Indus #5	Indus #6
Quality control of the surface prep	Only during the qualification phase Surface energy measurement (water contact angle)	No. In some cases , measurement of the surface energy during the qualification phase	No, with some exceptions (water test)
Step 3 : Bonding process			
Key parameters ?	A lot of thing are fixed (and listed into the process documentation) Cure cycle, parts and adhesive requirements, worker's habilitations....	A lot of thing are fixed (and listed into the process documentation) Cure cycle, parts and adhesive requirements, worker's habilitations....	A lot of thing are fixed (and listed into the process documentation) Bonding location, cure cycle, parts and adhesive requirements, worker's habilitations....
Environnemental condition	HR and T° measured with operation limit Clean room for some specific assembly	HR and T° measured with operation limit In some case, curing in an climatical chamber Clean room for some specific assembly	Temperature & Humidity are controlled (with limits) Additionnal rules for managing of co-activity (to reduce risk of pollution)
Curing process	Standard curing cycle given by the supplier In general, ambiant phase + post-curing in oven If to big, local heating (with local T° measurement during the qualification phase)	Curing cycle validated during the qualification phase by DSC measurement (can differ from supplier cycle) In oven, the position of the parts is fixed by procedures No measurement of the T° of the parts	Mostly curing at ambiant (large parts) but also in oven or auclave If autoclave, preliminatry thermic cartography taking account the number & position of the parts Curing % is checked during the qualification phase
Gap management	Gap is part of the definition of the bonding Gad managed by the toolings or using calibrated "fishing" line	By the volume of adhesive In a lot of cases, very low thichkness	Depending on the system : - by adding calibrated tissue - by design (mechanical blocking) - by the process (qty of adhesive, pressure)
Travelers or cut-off samples	Samples made for each adhesive mixing Mechanicaly tested (shear + traction) + 1 in aeging + 1 on spare	No in general In most cases, curing is followed on pure adhesive samples	- Adhesive sample for the hardness control - Less and less specimens for mechanical measurements
Statiscal analalysis	Yes, with a database of all results and analysis for detection of drift	No	For adhesive reception : control card For the process parameters : no systematic analysis
Step 4 : Quality control			
Systematic NDT	No In some case, measurement of dimension of the bonded zone	No For optical bonding, visual control	Ultrasonic scan quit systematic Shearography may also be used for bubble detection
Management of defects	??	On electronic cards, measurement of the bonding size with comparison with a refecence book	For each type of parts, a list of acceptable deffects is established (made in the beginning of the production phase) Defects could be justified by calcul and/or after complemtary NDT
First part qualification ?	No, only specific tests during qualification phase	Yes, trough validation of the global functionnement of the part - nothing dedicated to adhesion	Not systematic
Periodic sampling with destructive test	No	No	No

Questions	Indus#7
Quality control of the surface prep	None on metals Water drop test on composite
Step 3 : Bonding process	
Key parameters ?	A lot of thing are fixed (and listed into the process documentation) Bonding location, cure cycle, parts and adhesive requirements, worker's habilitations....
Environnemental condition	Temperature & Humidity are controlled, dust also
Curing process	Oven or autoclave Tools are fixed (but no thermic cartography) The cure cycle is validated and may be different from that of the supplier
Gap management	For film : via the pressure For paste : injection between the parts, until the cavity is filler
Travelers or cut-off samples	Systematic for 2K adhesives with in house mixing
Statiscal analalysis	Registred but not analyzed
Step 4 : Quality control	
Systematic NDT	Ultasonic scan systematic
Management of defects	For each type of parts, a list of acceptable deffects is established (made in the beginning of the production phase) Defects could be justified by calcul and/or after complemtary NDT Reparation is allowed
First part qualification ?	Yes
Periodic sampling with destructive test	No

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