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With the support of the FOT members

Acrobat Document

Pre –treatment for bonding Composites/Metal

FAA Bonded Structures Workshop
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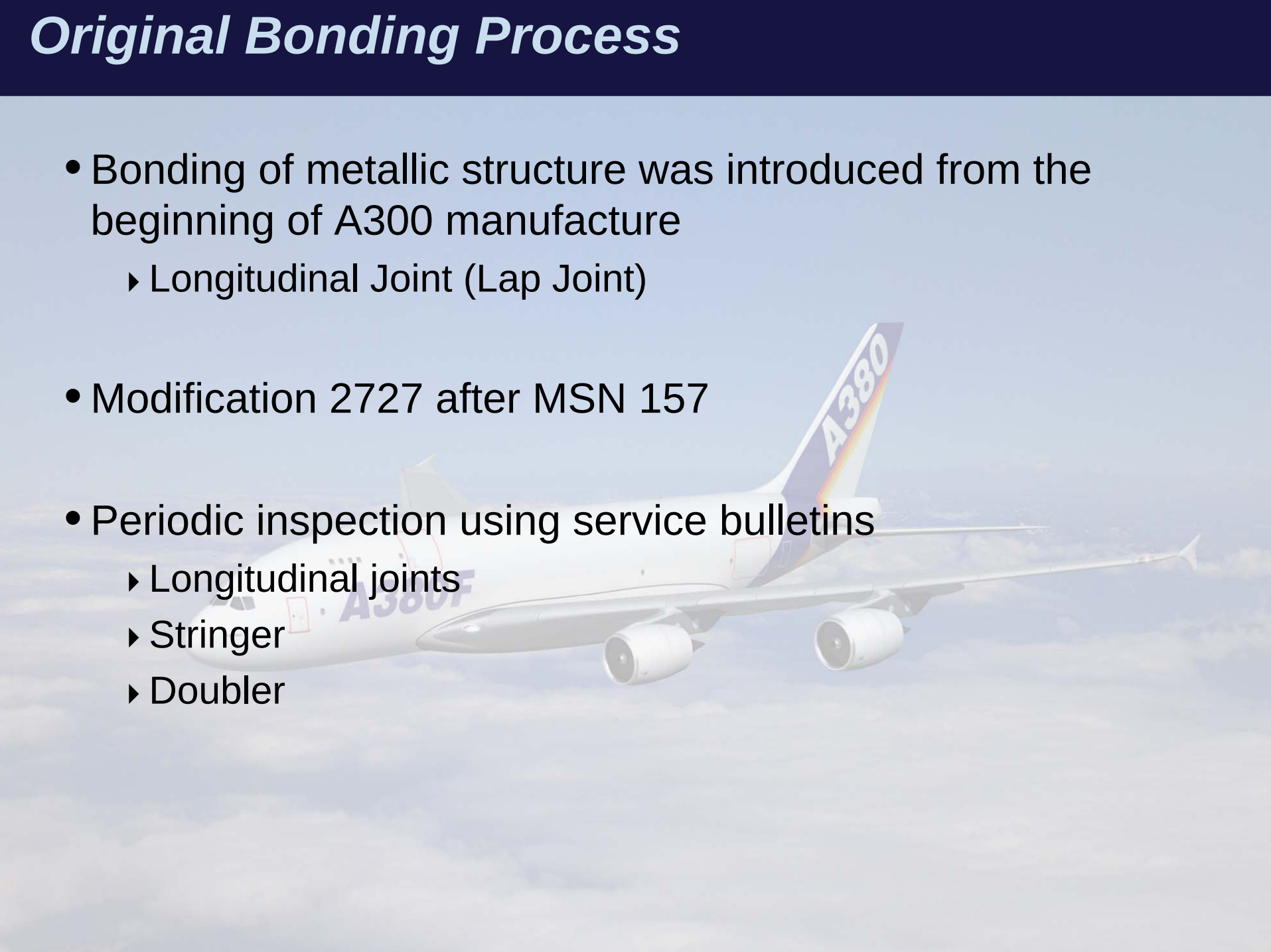
TOPICS

- Introduction
- Metal surface treatment
- Composite surface treatment
- Surface preparation evaluation
- Future Prospects

Original Bonding Process

- Bonding of metallic structure was introduced from the beginning of A300 manufacture
  - Longitudinal Joint (Lap Joint)
- Modification 2727 after MSN 157
- Periodic inspection using service bulletins
  - Longitudinal joints
  - Stringer
  - Doubler



History and Service Experience

- **Historical Causes of Failure**
  - Improper Surface treatment

  Failure mechanism was determined by Bondline Corrosion in both:
  - laboratory investigations and
  - Service Findings in affected aircrafts

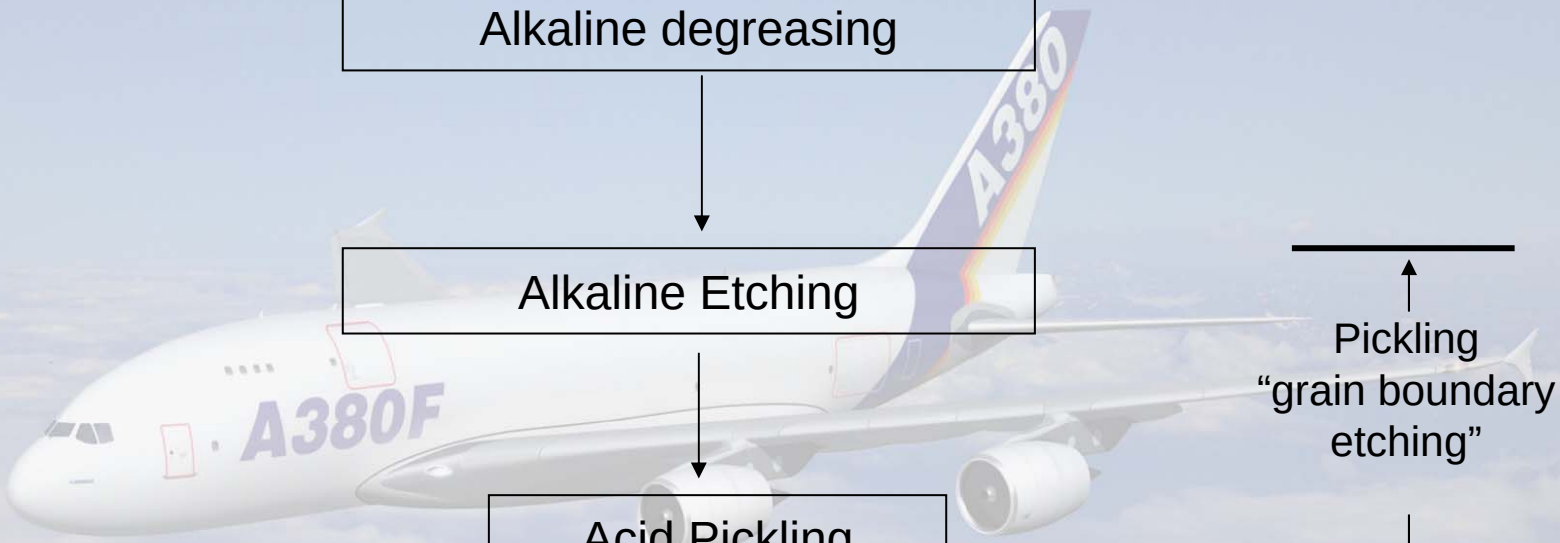
- **Corrective Actions**
  - CAA - end Pre-treatment
  - Advanced adhesive epoxy films + chromated primer
  - Design changes

State of the Art - Processes

For Bonding Application

1	Alkaline degreasing
2	Alkaline Etching
3	Acid Pickling
4	Chromic Acid Anodizing

Pickling
“grain boundary etching”



Acid Pickling Requirements

- **Applicable for Bonding processes**
- **Performance on all used Al-alloys nearly equivalent**
- **Activation of Al surfaces**
  - Removal of marking inks (still remaining after degreasing)
  - Removing oxide/hydroxide films
  - Desmutting properties
  - No significant roughening of surface; homogeneous, continuous appearance.
- **Only by chemical means, non electrolytic process**
- **Uniform, “pickling” topography**
- **Metal removal (constant ageing of the batch)**
- **Surface has to be compatible with post treatments**
- **No electrochemical deposition of Cu, Zn & Fe from the solution of the Al-surface**
- **No deposition of metal salts or hydroxides from the solution to the Al-surface**

Metal Removal before CAA

Bonding (NOR)		Metal removal
Alk. Cleaning	Metaclean T 2001	~ 0,25 µm
Alk. Pickling	Almeco 51	~ 1-3 µm
Acid Pickling	**CSA**	~ 2-4 µm
In total		**~ 4-6 µm**

Anodising Requirements (i)

- General Requirements
- Applicable for Bonding processes
- Oxide layer not to be easily damaged by manual handling
- No significant impact on damage tolerant behaviour
- No significant impact on fatigue behaviour
- Performance on all Al-alloys used in production
- Homogeneous oxide layer for all alloys, exeptional case: high Silicon containing alloys (>2%)
- Detectable, non contacted method should be mandatory

Anodising Requirements (ii)

Process Requirements

- Chemical solution (bath) should be stable for extended periods
- Quality easily controlled by defined levels

Oxide layer Requirements

- Hydrolysis products must have no deleterious effects
- Stability against manufacturing environment for defined time intervals
- Oxide layer covering the substrate completely with low electrical conductivity
- Compatibility with waterborne primers

Adhesion/Corrosion requirements

- No adhesion failure to substrate or primer
- Oxide layer must be as porous as possible
- Bondline-Corrosion resistant

Chromic Acid Anodising: Porous Structure

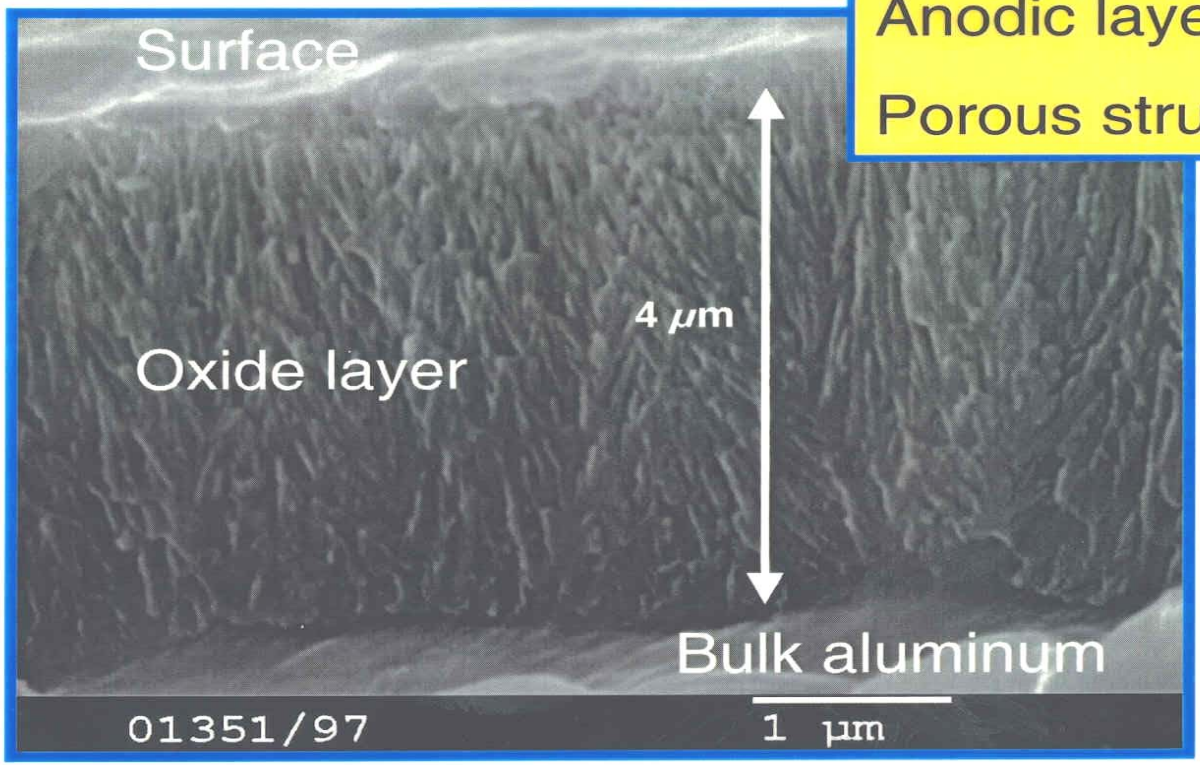
CAA: *40V, 25 min + 50V, 5 min Bengouth Cycle*

oxide layer with wide pores

sealable

good adhesion for bonding

good corrosion behaviour



Bonding-Primer (i)

- **Material Properties**
  - One component epoxy system
  - Low VOC (less 250g/l), 10 to 30% solid content
  - Not 100% water based (application aspects)
  - Containing corrosion inhibitors
  - No hazardous substances which require special protection
  - Coloured material for quality control purposes (thickness control)
  - Storage time at least 12 month at RT (or +4°C)
  - Work life at least 15 to 30 days (30°C/75% R.H.)
  - Compatible with all qualified structural bonding adhesive film or paste (use as standard primer)
  - Compatible also with 175°C curing adhesives and basic primer/top coats
  - Penetration of bonding primer in oxide layer (viscosity behaviour)

Bonding-Primer (ii)

- **Mechanical Properties**
  - Multiple curing (up to 5 times) without any degradation in performance
  - Primer layer thickness tolerance between 2 and 15 µm without degradating the quality of adhesive joint (e.g. peel load)
  - Fulfill the requirements AIMS 10-01-001 and 10-01-002 together with the appropriate adhesive film or paste.
  - Bondline corrosion: 90 days: ≤ 10%
180 days: ≤ 20%
300 days: ≤ 30%
  - Work life at least 15 to 30 days (30°C/75% R.H.)
  - Resistance to filliform corrosion (EN 3665)
  - Good adhesion on surface oxide layer

Critical Issues

- Replace current situation by a chromate free process
  - Pre-treatment
  - Primer
- What are the real criteria for implementation

Objectives

- Current projects intend to find systems for preparing the surface of carbon fibre composite elements before bonding.
- Especially focused on:
  - Structural bonding
  - Elements with big size areas to be bonded
  - Parts made off thermoplastic matrix composites
- The aim is to develop automated technologies
  - Reproducible
  - Accurate
  - Quality assessed
  - Easy to control and inspect
  - Ecologically favourable
  - Applicable for automation

Composite Surface Treatments

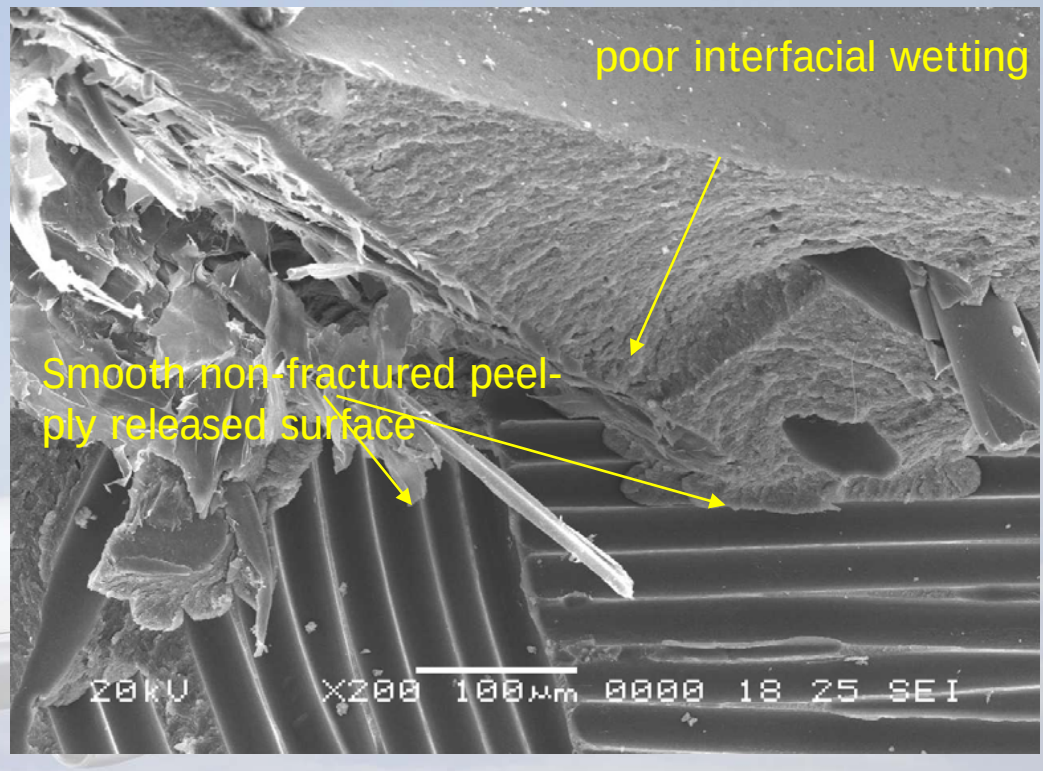
- Composite structures can have bonded joints fabricated by three different processes:
  - Secondary bonding
  - Co-bonding
  - Co-curing
- Surface preparation is a critical step in any bonded joint and must be clearly defined before any bonding is performed.
- Particularly important for:
  - Secondary and co-bonding processes.

Composite Surface Preparation Methods

- Peel ply
  - Dry
  - Wet
- Peel ply + additional surface preparation
- Plasma and Corona treatments
- Abrasion by means of blasting techniques

Peel Ply Surface Preparation

- Widely used within AIRBUS
- Probably the most effective and repeatable pre-treatment at the present time



Peel Ply Surface Preparation

- **Object**
  - Finding more efficient peel ply to be used as a pre-bond treatment without subsequent abrasion.
  - Understand factors affecting good or bad peel ply behaviour

- **Recent Works**
  - Investigation of different dry peel ply
    - Nature = Nylon / polyester
    - Different Weave patterns
    - Weave surface treatment
  - Evaluation of wet peel ply

⇨The best peel ply depends on the laminate type as well as the adhesive used.

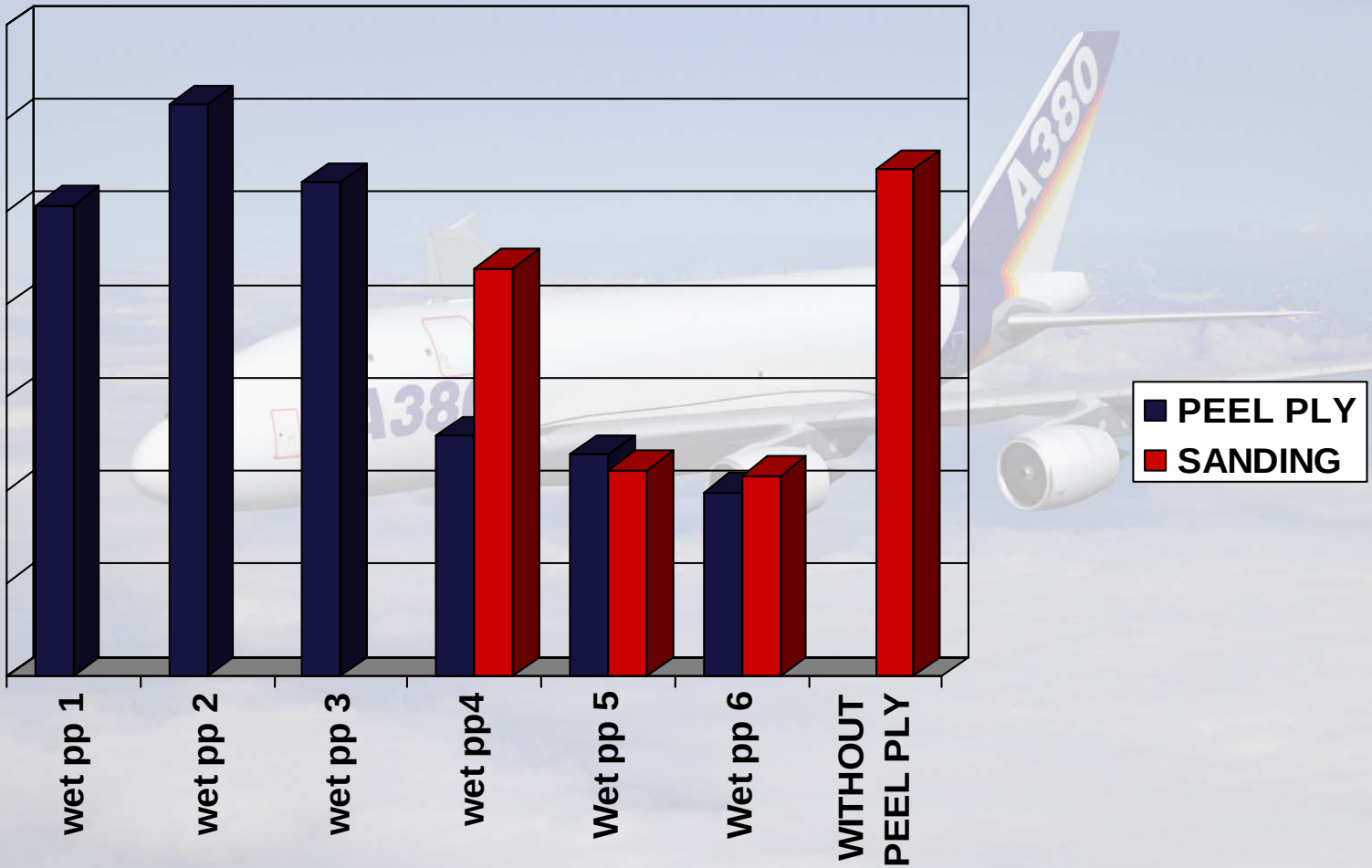
Composite Surface Treatments

WET PEEL PLIES

LAMINATE: 8552/AS-4 UD

ADHESIVE: FM 300 K .05

FRACTURE TOUGHNESS (G_{IC})



Plasma and Corona Treatments

- **Object**
  - Basic research on the effects of different types of source of plasma as well as corona discharge.
  - Mainly focused on the thermoplastic matrices composites.

- **Testing**
  - Shear strength Dry, Hot / Wet and exposure in hydraulic fluid.
  - Toughness (G_{IC}), Dry, Hot / Wet and exposure in hydraulic fluid.
  - Micro analytical investigations.

Plasma and Corona Treatments

- **Intermediate conclusions**
  - The study of different physical based pre-treatments showed for ND plasma a significant increase of shear strength as well as the fracture toughness energy after dry and hot / wet testing.

- **Further activities**
  - Analysis of the pre-treated surface.
  - Analysis of interphase.
  - Mechanical tests on the resulting bond line.

Abrasion of means of blasting

- **Object**
  - Define the best parameters to define a suitable and efficient pre-bond treatment by blasting techniques.

Preliminary Tests Conclusions

- **Treatment of carbon tape laminates**

  Dry blasting surface preparation increase the G_{IC} and SLS test results values
  Failure mode are predominantly cohesive.
  Using appropriate blasting parameters, carbon fibres of the laminates are not damaged.

- **Treatment of non-crimp fabric laminates**

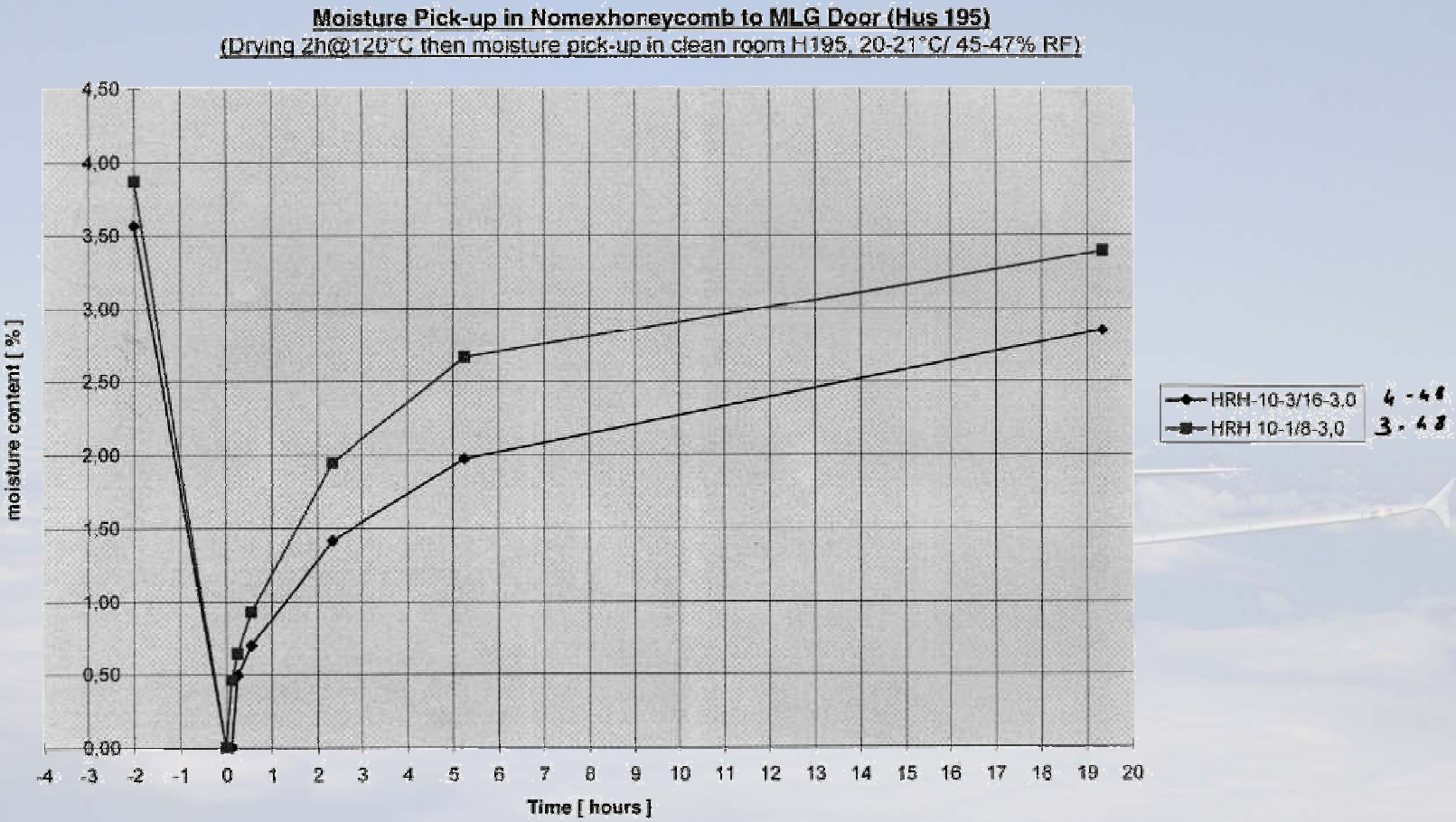
  First investigation confirms the same trend.
  Application of grid blasting increase the sear strength and the fracture toughness energy.

- **Further activities**
  - Investigations of different grid materials and processes variables (e.g. distance, angle) on different substrate has to be performed.
  - Optimum dry blasting parameters shall be determined in future tests.

Critical Issues

- Which criteria does the modified surface have to fulfill
- Do we have to require a type of failure mode ? A Measured strength
  or both ?
  - ⇨ How to predict long term durability with coupons
- Any change must be checked by additional testing e.g. extensive compatibility tests.
- Determination of release test to monitor batch to batch consistency.
- Define storage condition for laminate as well process step durations' (e.g. assembly time).
- Study effects of pre-bond humidity.
  - Laminate
  - Honeycomb

Moisture pick-up in Nomex honeycomb



Surface Preparation Evaluation

- Deep”academic” work has been performed regarding test methods for adhesives and adhesive joints that could eventually be applied to assess the surface preparation quality of composite adherents for bonded,
  co-bonded and co-cured composite joints.

- There is very little published scientific work concerning the evaluation of adhesive joints between composite materials.

- Most of the existing work is related to standards or international committees for standardization.

Mechanical test methods

- Peel stress test methods are more sensitive to surface preparation than shear stress methods.
- Actually the most sensitive existing test method still seems to be the G_{IC} DCB test in all its different configurations.
- G_{IC} DCB test method is expensive, difficult to prepare and test correctly.

- **Final conclusion**
  - Existing test methods do not fit exactly with the requirements demanded.
  - For composite it’s necessary to develop a new “ simple and reproducible” test to evaluate the quality of the structural bonding.

Physical test methods

- No direct correlation has been found between percentage of Si and mechanical behaviour of the bond line by means of G_{IC}.
- No direct correlation has been found between percentage of F and mechanical behaviour of the bonding line by means of G_{IC} in the percentages found in the laminate.
- Direct correlation has been found at higher values of F content (due to other ancillaries contamination).
- Direct correlation has been found between failure mode and extractable residue.

Future Prospects

- Surface preparation
  - Metal: substitution of chromate (Surface treatment + primer)
  - Composite: enhancement to peel ply or replacement
- Adhesive
  - Stability in out time and pre-bond humidity
- Inspection
  - NDI for weak interface detection
  - Strong request to define the equivalent to the wedge test for composite.

THANK YOU FOR YOUR ATTENTION

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