

## Adhesion of aluminium alloys : morphology, surface chemistry and adhesive bond durability

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### Introduction

For many years the aerospace industry has been using adhesive bonding for a variety of applications. To that way, chromic acid anodizing of aluminium is an effective process for producing a substrate (porous oxide film) which provides excellent surface layer properties prior to assembly, mainly through epoxy based adhesive [1]. However, recently the use of metal finishing treatments involving hexavalent chromium has become restricted because of environmental consideration.

Consequently, among the different goals to be reached by the aeronautic industry, particular efforts are in progress that aim to propose innovative processes exempt from unsuitable species such as hexavalent chromium.

Keywords : Adhesion, Coupling agent, Wedge test, Polymer-metal Interface,

### Experimental

Aluminium surface specimen were first degreased and cleaned prior to any further treatment in order to remove both pollutants and naturally formed external oxide layer.

Then, a variety of innovative cleansing and/or etching surface treatments was investigated in order to induce specific morphology and chemical characteristics.

**Morphology** was investigated through Scanning Electron Microscopy equipped with Energy Dispersive X-ray analysis (SEM-EDX) or Field Emission electron microscopy (SEM-FEG) depending on the information to be reached.

**Chemical analysis** was performed using Glow discharge spectroscopy (GDS) thus allowing to get a depth analysis of the metal extreme surface prior and after treatment.

**Mechanical experiments** were investigated using wedge test [2] standardized method (ASTM D3762-79, 1993) consisting in first evaluating the initial crack extend at equilibrium (room conditions) and secondly monitoring the crack propagation over time when specimen assembly are exposed to hydrothermal ageing in an environment chamber (50°C, 96% Room Humidity). Figure 1 and figure 2 illustrate the wedge principle and a typical crack propagation profile over time respectively.

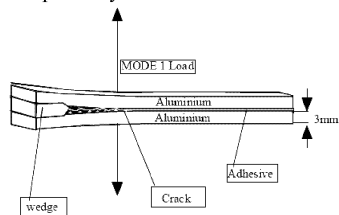


Figure 1. Wedge test principle [reprinted from [3]]

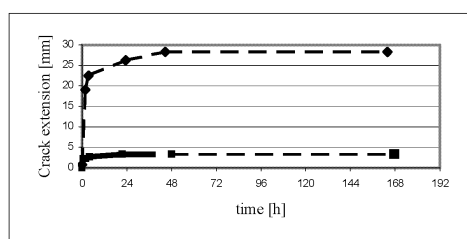


Figure 2. Typical crack propagation for an aluminium joint submitted to hydrothermal ageing conditions (■: good ◆:fair)

### Results and discussion

Successful bonding requires a clean and chemically active metal surface. The strength and durability of the joint depends upon chemical and micro-mechanical interactions at the surface, involving, among other surface characteristics, metal's porosity and microstructure (as illustrated by figure 3) as well as susceptibility for primer and/or adhesive to form covalent bonds together with the surface.

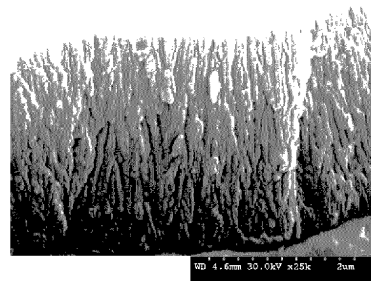


Figure 3. Aluminium oxide morphology and micro-porosity as evidenced by Electron Microscopy of aluminium showing

The present study deals with chromium free experimental conditions for both deoxidizing and anodizing treatments, and have been evaluated and compared to classical undesirable conventional treatment conditions (sulfo-chromic etching and Chromic Acid Anodizing treatment).

In addition, primer agents such as organosilane have been tested as an alternative to strontium chromate primer as a potential environmental-friendly adhesion improver [4].

Data collected have been used to calculate crack growth as function of time for the bonded specimen and resulting failed surface have been examined to establish failure locus. As illustrated by figure 4, unsuitable behavior correspond to interfacial failure (top) while the best assembly properties can be obtained as the failure become cohesive inside the adhesive layer (bottom).

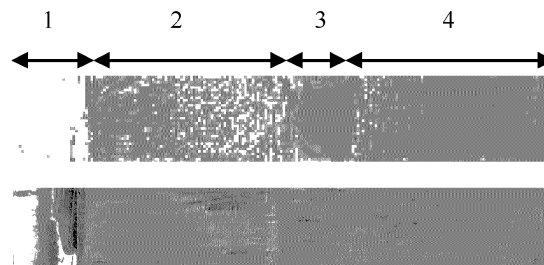


Figure 4. Failure profile as examined in case of interfacial (top) and cohesive (bottom) rupture modes

Legend: 1 Initial wedge insertion length at 23°C  
2 Initial crack propagation at 23°C for 2 hours  
3 specimen left at 96% RH at 50°C  
4 Specimen opened at 23°C

### Conclusions

The whole results allow to demonstrate high potentiality for environmentally friendly treatments of aluminum substrates as some of the treatments tested allowed to get suitable metal-adhesive. In particular, the role of both metal surface porosity and chemical composition has been highlighted thus allowing to propose alternative to classical treatments for aeronautic applications.

### References

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