# STRUCTURAL ADHESIVE BONDING IN AEROSPACE APPLICATIONS

EDUARDO A. S. MARQUES RICARDO J. C. CARBAS ALIREZA AKHAVAN-SAFAR A. FRANCISCO G. TENREIRO LUCAS F. M. DA SILVA AUTHORS

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#### TITLE

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### PREFACE

Adhesive bonding has been around for thousands of years, dating from pre-historic times. However, it is the aerospace sector that pioneered this technology in industrial terms. That's because adhesively bonded structures are lighter than structures built using traditional joining methods. Since the First World War with wooden aircraft, the aeronautical industry has shown the way to other vehicle structures and nowadays adhesives are used in many transport structures such as automotive, railway or marine.

This short book intends to give the fundamentals of adhesive bonding with aerospace applications in mind. Whenever possible, the basics are related and interpreted with aeronautical cases. The adhesives typically used in the aircraft sector, the surface preparation of the materials used such as aluminium or composites, the design of joints typically seen in plane structures, the fabrication methods and durability issue such as fatigue are treated. The text is very easy to follow and accompanied by many illustrations to facilitate the comprehension of the concepts exposed.

The depth of this book is sufficient for master students that are focused on aeronautical issues and give a first insight of the technology. It does not substitute reference books on the subject, but it is a good introduction for further research studies.

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### INTRODUCTION

### 1. INTRODUCTION

Joining methods are a set of technologies that allow two or more components to be combined, creating novel structures which are more complex and varied in material composition and that cannot be obtained via a direct manufacturing process.

Although many different joining processes are available to an aeronautical designer, such as the use of fasteners, welding, clinching or riveting, the use of adhesive bonding in aircraft construction facilitates the manufacture of lightweight structures, enabling the use of materials which are unsuitable for use with these other classic joining methods. Adhesive bonding has always played a role in aircraft construction but nowadays it is increasingly common to see manufacturing solutions whose existence is only possible with the use of adhesives. A classic example of such products is the modern composite airliner, whose complex shape and construction, combining different composites and metal composites, is the product of an extensive use of structural adhesive joints.

As we will see throughout this book, the study of adhesive joints and the phenomenon of adhesion is a complex science which requires a multidisciplinary knowledge of physics, chemistry and mechanics. This is because adhesive bonding often requires mastery of a wide range of processes such as joint design, surface preparation and the proper selection and modification of base materials. In an adhesive joint, the key component is obviously the adhesive, a material that, when applied to surfaces of other materials, establishes a physical link between them, capable of resisting separation forces. However, it is crucial to stress that adhesives do not all behave in the same way. One simple and easy way to differentiate between them is to create a boundary between structural and non-structural adhesives. By convention, a structural adhesive is thus said to be an adhesive that can resist substantial loads and that will be responsible for the strength and rigidity of the structure. It is customary to classify as structural adhesives those that have a shear strength greater than 7 MPa. Furthermore, a structural adhesive joint is also one that is stable throughout the useful life of the structure. In aeronautical construction, we can typically find structural adhesives bonding secondary flight structures, fairings and interior fittings. Despite their lower strength, non-structural adhesives are also of great importance. These include, for example, the sealants which line fuel tanks and fuselages and are essential to the construction of gas-turbines, providing sealing of high temperatures gases.

### **1.1. BASIC ADHESIVE BONDING CONCEPTS**

Before we delve deeper into a detailed analysis of the different aspects of adhesive bonding in aerospace construction, it is first necessary to precisely define the most important concepts associated to this process and know the correct terminology. Simply put, an adhesive is the substance that initially fills the gap between the materials to be bonded, which adheres and solidifies to them. When the application is of a non-structural nature, the adhesive is usually known as a sealant, which indicates a function that is more oriented towards gas or liquid sealing and less suitable for load bearing (although it can still do so). The materials to be bonded are called substrates and after bonding they are called adherends, although in practice one might find that these two terms are sometimes used interchangeably. Between the adhesive and the adherend, an interface is always found, corresponding to the contact plane between the surface of the two materials (as shown in Figure 1.1). This interface is a complex region, with great importance in the joint performance and durability.





Adhesives operate by exploiting adhesion phenomena. Adhesion will be described in further detail in Chapter 2 of this book, but for now we can define it as the process of attraction between two substances, resulting from intermolecular forces established between them. Inside the adhesive and the adherend, we find only the cohesion forces, defined by the intrinsic properties of the materials. A completed adhesive joint is therefore formed by the adhesive, the adherends and the interface, but it may also include intermediate layers of different functions, such as primers or coatings and even reinforcements such as fibres and particles.

The ultimate goal of a joint designer, be it in the aerospace sector in any other industrial field, should be to ensure that the adherend is always the weakest link of the joint. In other words, one should aim for a condition where the presence of the joint does not weaken the strength of the structure that contains it.

### 1.2. A BRIEF HISTORY OF BONDING IN THE AERONAUTICAL INDUSTRY

Throughout human history, adhesives have always played an important supporting role, encompassing multiple uses. In fact, archaeological evidence suggests that 200,000 years ago Neanderthals used a tar-based adhesive to make axes and spears. Many millennia later adhesives first became intertwined with mankind's desire to be able to fly, featuring in the myth of lcarus and Daedalus (Figure 1.2). In this story, the father and son team created wings, by gluing feathers with wax, to escape from Crete, but those of lcarus ultimately failed when exposed to the heat generated by the sun when flying too high and the humidity of the sea when flying too low (what we would call today a durability problem).



Figure 1.2. The flight of Icarus (Jacob Peter Gouwy, 1637).

The start of the 20<sup>th</sup> century corresponded to the birth of modern aviation, which quickly adopted the use of adhesives. The Wright brothers used horsehide glue; an organic colloid of protein derivation found in the hide of horses to assemble their historic Flyer. And little more than a decade later, aircraft were no longer flimsy contraptions but fully fledged war machines. During the First World War adhesives were extensively used to bond the structures and coatings of military aircraft (Figure 1.3). However, these adhesives were still mainly based in naturally sourced materials. Casein glue, obtained from milk proteins, was the preferred adhesive for these applications since it was relatively more durable to moisture than glues sources from animal hides.



Figure 1.3. Aircraft of bonded wooden construction used during World War I.

The formulation of adhesives from petroleum derivatives revolutionised both the versatility and capabilities of adhesives. Between 1920 and 1940, significant progress was made in this area, but the use of adhesives in aeronautical applications was still regarded with some reluctance, especially in an era where aircraft almost universally adopted a metallic construction, being joined with rivets.

A major revolution in the application and capabilities of adhesives occurred during World War II, where new synthetic adhesives were formulated for use in the aircraft industry. Aircraft and other military equipment were produced at a frantic pace and the challenging performance demands produced forced the implementation of new structural bonding methodologies. The deHavilland Mosquito, a British light bomber, is one of the most important examples (Figure 1.4). Adopting a fully wooden construction, using pre-formed plywood components to minimize the usage of scarce and strategic metals, the Mosquito used urea formaldehyde resin adhesives to bond together its wooden structure. The combination of the bonded lightweight construction and powerful engines allowed the Mosquito to be one of the fastest aircraft of WW2.



Figure 1.4. The deHavilland Mosquito, a World War II high performance aircraft built of wood and bonded with structural adhesives.

The adhesives used in the deHavilland Mosquito were still quite brittle and were quickly replaced by new formulations developed by N. A. de Bruyne and his team at Duxford, Cambridge, combining polyvinyl formal and phenolic resols to achieve much improved toughness. This product was a quantum leap in adhesive technology, and it is still used today (more information on this adhesive is provided in Chapter 4). Later on, in the 1950s, this technology was expanded to bond metallic structures, playing a key role in the manufacture of the deHavilland Comet jet airliner (Figure 1.5). The partially bonded construction of the all-metal fuselage and wings resulted in high stiffness and strength, coupled with a relatively low weight.



Figure 1.5. The deHavilland Comet, the first jet airliner to reach service.

Since then, the use of adhesives was extended to a myriad of structural and non-structural applications, hinged on a constant development of new adhesives and bonding processes that has never slowed down. Today, this joining technology is highly mature, safe and efficient, being a key process for the construction of high-performance aircraft.

### 1.3. MAIN ADVANTAGES OF ADHESIVE BONDING IN AEROSPACE APPLICATIONS

Adhesive joints are known to possess a very peculiar set of characteristics that distinguish them from other conventional joining methods, such as welding, fastening and riveting.

#### 1.3.1. Key advantages

When properly designed and implemented, bonded joints can provide a very uniform distribution of stresses, reducing stress concentrations. The use of adhesives naturally avoids highly localized, point to point connections that generate a high stress level in the joined materials, since the resultant connection is practically continuous, as shown in Figure 1.6.



Figure 1.6. Stress fields obtained with three different joining methods. Welding (left), riveting (centre) and adhesive bonding (right).

It is this uniform stress distribution that provides adhesive joints with an excellent fatigue resistance. Figure 1.7 clearly demonstrates that an adhesive joint can have a fatigue performance close to that exhibited by the base metal, something that is never possible with riveted joints, since it requires drilling (and thus damaging) the adherend material.



Figure 1.7. Comparison of the fatigue performance of two joining methods (bonding and riveting) and the base material.

Helicopter blades are mainly of bonded construction since the large bonded areas provide low stress concentrations, critical for these structures which are subjected to highly cyclical loads.

Furthermore, adhesives allow the manufacture of light structures without additional elements such as screws and weld beads and confer increased flexibility to the design and manufacturing processes, being easily automated. They are also able to join dissimilar materials with relative ease, and this includes materials of very different nature (e.g. when joining metals and composites in an airframe). It is this flexibility that enables the creation of the highly optimized multimaterial structures that we nowadays find in aerospace construction, which explore the advantages of composites, metals and polymers. One classical example of these structures are honeycomb panels, where low density cores (of metallic or polymeric nature) are combined with stiff metallic or composite skins.

Adhesive joints can also be easily employed for large bonding areas, improving stress distribution and structural stiffness with limited implications for the cost or simplicity of the process. They exhibit good vibration damping properties and act as seals, allowing to combine the bonding and sealing roles in a single layer. They do not require drilling holes or other geometric changes to the adherends, a feature that is especially useful for joining composite materials, as these lose most of their strength when modified in this way. Using adhesive joints also allows to avoid galvanic corrosion processes and enhance electrical and thermal insulation, as the adhesive layer ensures that there is no direct contact between the parts to be bonded.

The aerodynamical qualities of bonded fuselages are also superior to those obtained with riveting, since the outer surfaces of the joints are much smoother and completely devoid of discontinuities resulting from the presence of rivets, fasteners or welding beads. Although the use of flush rivets can in part compensate for this difference, these come with a much higher process cost.

#### 1.3.2. Limitations

Some important limitations are associated to the use of adhesive joints. One of the most important is certainly the indispensable requirement for a careful and appropriate surface treatment preparation, especially for bonding polymeric and composite substrates. As we will see in Chapter 2 and Chapter 3, an incorrect surface preparation can have a major effect on joint strength and cause premature failure of the joint.

Another important limitation of adhesive bonding is related to its sensitivity to the loading direction. Adhesive joining performs very well for shear loads but exhibits low resistance to peel and cleavage stresses. These loading modes are shown schematically in Figure 1.8.



Figure 1.8. Peel, cleavage and shear loads in adhesive joints.

Cleavage occurs when the load is concentrated on one end of the joint, while the opposite side remains effectively unstressed. In essence, this type of load pries the joint open, almost as if a lever was applied at the end of the adhesive layer. Consequently, the stresses acting on the adhesive are maximum near the area where the cleavage load is being applied and minimum at the opposite end of the joint. It is this concentration of stresses that results in a very low resistance to cleavage.

Peel loads are concentrated along a thin line at the edge of the adhesive layer and occur mainly when at least one adherend is very flexible. These are the most damaging loads that an adhesive may be subjected to. In this case, the load we are applying is concentrated in an extremely small area, which explains the limited resistance offered by the adhesive and, consequently, by the joint. In joint design, it is essential to use geometries that avoid localised stresses and guarantee a relatively uniform stress distribution such as those attained during shearing. If the loads are not perfectly parallel to the adhesive layer, significant peel or cleavage forces may arise, severely limiting joint performance.

One of the greatest challenges associated with the use of adhesive joints in safety critical applications, such as aircraft construction, is the low resistance of adhesives to extreme environmental conditions. In many cases, adhesives, as all polymeric materials do, exhibit an important variation of their mechanical properties as a function of exposure to environmental conditions. To properly design an aerospace structure that will see service under such conditions, detailed characterisation and validation tests are indispensable, allowing to quantify the reduction in joint performance.

Still in the subject of joint design, one important issue associated with adhesive joining is the lack of a widely accepted universal criteria for the determination of joint strength. The vast diversity of methods for joint design, with different characteristics, capabilities, and limitations, might represent too large a challenge for those who are inexperienced with this joining technique.

Large-scale, high-volume manufacture of bonded structures also creates some important challenges, such as those associated with handling the structures immediately after the application of the adhesive. In practice, most bonded joints are not immediately ready to be handled after manufacture, which can delay some production processes. As such, there are also special fixing requirements that are critical to hold the parts together during the hardening process, requiring the investment in moulds and jigs, as well as the implementation of procedures which accelerate adhesive hardening. Difficulties are also associated to the disassembly of glued parts, which creates challenges in both the repair and recyclability of aspects of these structures.

Lastly, the use of adhesive bonding in the aeronautical industry is also hindered by the fact that the quality control and non-destructive testing of bonded structures is very complex. As we will see in the Chapter 6 of this book, although many technologies can be used to detect voids, crack and other large defects, no technique exists that can reliably detect weak adhesion. This represents a case where the adhesive is touching the adherend, but there is no load carrying capability, since adhesion is practically inexistant. The lack of a method suitable for detecting this major, yet subtle defect, places important limitations to the use of adhesives in primary flight structures, as regulatory bodies are naturally comfortable with the possibility of aircraft flying with completely unseen defects in the bonded joints. Thus,

adhesive bonding is still majorly used alone solely in secondary flight structures, those that do not risk the integrity of the complete vehicle in case of failure.

### 1.4. EXAMPLES ADHESIVE BONDING IN AEROSPACE APPLICATIONS

#### 1.4.1. Aircraft and rotorcraft

As stated beforehand, riveting was the method of choice for aircraft construction for most of the 20<sup>th</sup> century, representing a fast, cheap and effective technique for joining and one that can be easily automated. Unlike welding, this process allows for joining practically all types of materials and can combine materials of dissimilar nature in the same joint. However, riveting requires drilling a large number of holes in the base material, a source of severe stress concentrations. Furthermore, it requires the use of seals to ensure tightness and the presence of exposed rivet heads can also be detrimental to the aerodynamic qualities of the aircraft, requiring the use of more expensive and less flush rivets.

Thus, the use of adhesives in aerostructures has increased exponentially, allowing for different types of materials to be joined, doing so without introducing large thermal stresses, free of holes and other geometrical modifications. Adhesives can be found in various locations in an aircraft (Figure 1.9), from structural components (mainly in secondary structures) to interiors, engine housings and other external structures such as covers and fairings. Inside an aircraft, adhesives can be found in cabin framing materials, in seating and used in thermal and acoustic control systems.



Wings and fuel tanks

Figure 1.9. Main areas of adhesive use in a modern aircraft.

A key structural use of adhesives in aerospace construction is in the manufacture of very rigid and lightweight sandwich panels, an application where adhesives (as shown in Figure 1.10) are used for bonding lightweight (and mostly hollow) cores to high strength skins.



Figure 1.10. Typical components of a sandwich structure manufactured using bonded construction.

Sandwich structures use very thin sheets in the skins, avoiding buckling with the use of bonded lightweight cores, creating a separation between the two skins. The strong and thin skins will mainly carry the tensile and compression stresses, while the honeycomb (and the adhesive which connects it to the skins) will be able to support shearing stresses. The most important advantage of a sandwich structure is the fact that it can provide exceptionally large stiffness with a very limited weight penalty, as shown in Figure 1.11.



Figure 1.11. Influence of core thickness in the performance of a sandwich structure.

Sandwich panels can be composed of a wide variety of material combinations and skins and core designs. The skin can be of aluminium alloys or composite, using Kevlar, carbon or glass fibre reinforcement. The core materials can be aluminium honeycombs, polymer and metal foams, very light composites or even preformed structures, such as pultruded composite profiles. Sandwich structures are also essential to dampen engine noise, in the form of acoustic panels, and to contain debris from catastrophic jet engine failures.

Modern rotorcraft are mostly composed of composite materials (such as carbon, glass and aramid fibre composites), chosen with the main purpose to reduce weight, dampen vibration

and consequently improve performance and fuel consumption. This type of composite heavy construction relies greatly on adhesive bonding, dispensing fatigue prone fastening and riveting whenever possible.

The bonded components present in a helicopter are varied and include, for example, the main and tail rotor blades, cabin floors and doors, fairings and aerodynamic stabilizers. Since many of these components present quite flat bonding surfaces, film adhesives are extensively used, providing good design tolerances and easing the manufacturing process (Figure 1.12).



Figure 1.12. Main areas of adhesive use in a modern helicopter.

#### 1.4.2. Space applications

The space sector makes extensive use of adhesive bonding in the construction of launch vehicles and satellites. As is the case for many other sectors, the use of adhesive bonding in these applications is strongly intertwined with that of composite materials. While at the beginning of the space race both launchers and satellites were mainly made of metal, composite monolithic structural parts started to become the norm in the 1970s, replacing many (but not all) metallic structures. This use was due to the association of epoxy resin with glass and boron fibres, which led to improved strength and stability to composite, although its use was with still limited to secondary structures such as fairings and supports. In the 1980s, the use of carbon fibre started to become the norm and opened the use of composites in primary structural parts, in monolithic structures or sandwich panels. Nowadays, many rockets include fully adhesively bonded composite stages which serve as tanks, holding propellant cooled to cryogenic temperatures. Some of these applications are shown in Figure 1.13.



Figure 1.13. Main areas of adhesive use in space applications.

Adhesives are used not only for assembling the structure of these vehicles, but they also find use in other more particular applications, such as fixation for accessories, sensors and thermal insulation. One of the most well-known of these applications is in thermal protection panels, where the excellent thermal resistance and flexibility of silicone-based adhesives make it possible to withstand the high temperatures of atmospheric re-entry and to accommodate the resulting large volume expansion differentials. Figure 1.14 shows the bonding system used to connect ceramic tiles to the aluminium skin of the now retired space shuttle orbiter. A room temperature vulcanizing silicone was used to both fill the surface gaps and to bond the tiles.



Figure 1.14. Schematic of the construction of a bonded heatshield for aerospace applications, highlighting the use of a room temperature vulcanizing silicone.

However, there are still some important limitations associated with the use of adhesives in aerospace applications. For example, as adhesive joints are highly sensitive to contamination, it is essential to ensure a clean and inert room during adhesive application. The issue of outgassing is also of importance. When subjected to a vacuum, some adhesives might release trapped gases, which can migrate to the multiple delicate sensors present in probes and satellites and contaminate them. Thus, adhesives intended for these applications must first be subjected to a battery of vacuum tests.

An additional challenge related to the use of adhesives is the limited knowledge of the long-term performance in outer space conditions, especially considering the extreme temperature gradients and incident radiation.

## THEORY OF ADHESION

## 2. THEORY OF ADHESION

At first glance, adhesive bonding process seems a quite simple and straightforward process, with the adhesive creating a strong and perfect connection to the adherends and providing a path for load transfer. However, this is not always true and limited adhesion can exist between the adhesive and the adherend, which will cause the load-bearing capacity to be very low, leading to premature failure of the bonded structure. The adhesion phenomena involved in the bonding process are quite complex and it is important to have at least a basic understanding of the mechanisms involved to be able to design and engineer strong and durable joints.

### 2.1. ADHESION FORCES

We can start our study of the adhesion forces by visualizing bonded joints as simple bridges between patch surfaces. There are two essential types of forces at play in this model. The first of these forces is adhesion - the bonding force between the adhesive and the adherend. The second is cohesion - the internal force of the adhesive and the adherend. Failure of an adhesive joint can occur in three possible ways, as illustrated in Figure 2.1. These are adhesive failure (failure at the interface), cohesive failure (which can occur within the bulk of the adhesive or the adherend) and finally mixed failure, a combination of adhesive and cohesive failure on the same fracture surface.

Failure modes



Figure 2.1. Typical failure modes in bonded joints.

It is at the interface that adhesives bond to adherends, forming chemical bonds. The forces involved depend on the chemical nature of the surfaces of the materials in question. The bonds that can be formed are divided into primary and secondary bonds, as seen in Figure 2.2.



Figure 2.2. Types of bonding forces acting in adhesive joining.

Primary bonds are those where there is sharing or transfer of electrons between the atoms involved and include ionic, covalent and metallic bonds. They are very strong bonds (100-1000 kJ/mol) but operate over very short distances, being effective only over a few angstroms of distance (10<sup>-10</sup> m). In contrast, secondary bonds do not depend on electron sharing or transfer, but rather on the interaction between atomic and molecular dipoles. These include van der Waals forces and hydrogen bonds. These are capable of acting over relatively long distances but are much weaker (<100 kJ/mol).

Figure 2.3 shows how the energy of these primary and secondary forces varies with distance.



Figure 2.3. Bonding energy of different types of forces acting on the adhesion process.

#### 2.2. SURFACE WETTING

In addition to the bonding forces present, another important factor for ensuring a good level of adhesion is the quality of the wetting of the substrate achieved by the adhesive.

One of the parameters that most influences wetting is the roughness of a surface. However, there is no universal optimum value for roughness and, consequently, it becomes necessary to adapt the surface preparation to the material that will be bonded, while taking into account the nature of the adhesive. If an adhesive spreads perfectly on the surface and if intimate contact can be achieved between all surfaces to be bonded, chemical bonds can now act on all parts of the surface, even in peaks in rugosity which are far apart from each other. Of course, spreading will be easier if the adhesive is liquid and if it can flow freely along the entire rough surface, filling all cavities as illustrated in Figure 2.4.



Figure 2.4. How adhesive can fill the gaps between two highly irregular surfaces.

Once wetting has occurred, hardening of the adhesive will take place and a connection between the adhesive and the adherend will be established (Figure 2.5). It should be noted that increased surface roughness influences wettability. Surface roughness can be modified through surface preparation, and this is one of the most delicate and important operations in joint fabrication. This topic will be discussed in more depth in Chapter 3.



Figure 2.5. Wetting of a surface during adhesive application and hardening.

In an ideal scenario, liquid adhesives would always be able to completely wet all surfaces to be bonded, but this does not always occur. In fact, in many cases the adhesive is effectively repelled by the surface. An analogous phenomenon can be seen on the leaves of certain plants when these become wet. Instead of creating a uniform and well-spread film on the leaf surface, the water instead takes the form of many individual droplets, as shown in Figure 2.6. This phenomenon occurs because the surface is hydrophobic, repelling water molecules. Hydrophilic surfaces, in contrast, attract water molecules and generally result in much better wetting by an adhesive.



Figure 2.6. Water drops being repelled by a leaf.

In a liquid, as is the case for an uncured adhesive, the forces of attraction between the molecules are in a condition of equilibrium in all directions. On the surface of the liquid this kind of equilibrium cannot exist because there are no neighbouring molecules outside the surface. These molecules are subjected only to a force which pulls them into the liquid (see Figure 2.7). To bring new molecules to the surface, additional work must be done and thus the molecules at the surface must have a higher energy than those on inside the liquid. This extra energy of the molecules at the surface is called surface free energy or simply surface energy, expressed as energy per unit area (with units mJ m<sup>-2</sup>). More precisely, this corresponds to the energy required to create one unit area on a surface.



Figure 2.7. Air/liquid equilibrium at the surface of a fluid.

The minimum energy principle is used to determine whether or not a liquid spreads on a solid surface. This principle explains why a liquid spreads on a solid with a higher surface energy but not on one with low surface energy. If the high energy solid surface is replaced by a lower energy surface (the liquid), then the total energy of the system is reduced and this will be a spontaneous process.

To summarize, it can be stated that:

- Energy of the liquid  $(\gamma_1)$  < Energy of the solid  $(\gamma_s)$  the liquid spreads
- Energy of the liquid  $(\gamma_1)$  < Energy of the solid  $(\gamma_c)$  the liquid does not spread

High energy surfaces are those where  $\gamma_s$  varies between 500 and 5000 mJ m<sup>-2</sup>, corresponding, for example, to metals and their oxides, relatively hard materials with high melting points. Low energy surfaces, on the other hand, have a  $\gamma_s$  between 5 and 100 mJ m<sup>-2</sup>; these include most polymeric (and composite) materials.

The contact angle between the liquid droplet and the solid surface provides an inverse measure of wettability and allows the surface energy to be determined using Young's equation. The angle formed by the solid surface and the tangent line to the upper surface at the end point is called the contact angle, as illustrated in Figure 2.8.



Figure 2.8. Contact angle formed by liquid drops in the surface of a liquid and Young's equation which balances the tensions present.

### 2.3. THEORIES OF ADHESION

A set of diverse theories has been proposed to explain the phenomenon of adhesion using single mechanisms. These can be seen only as partial explanations, since adhesion is in most cases the result of a variety of mechanical, physical and chemical forces interacting with each other. These forces include mechanical interlocking, which is caused by mechanical anchoring of the adhesive to the surface topography, electrostatic forces and the intermolecular and chemical forces that exist in the interface zone. The theories that explain the existence of these forces are the mechanical theory, adsorption theory, diffusion theory and electrostatic theory. The mechanical and adsorption theories are described in detail in the following sub-sections.

#### 2.3.1. Mechanical theory

According to the mechanical theory, adhesion occurs if there is a certain level of irregularities on the surface, resulting from roughness or porosity. A mechanical interlocking process occurs when the liquid adhesive penetrates and fills all cavities and irregularities of the adherend and subsequently solidifies, as shown in Figure 2.9.



Figure 2.9. Mechanical interlocking between the adherend and the adhesive.

This theory assumes that the adhesive is able to wet the adherend thoroughly and has adequate flow capabilities (rheological properties) to penetrate the fine pores and voids quickly. Please not that although this is an important mechanism in the formation of a strong bonded joint, in many cases good adhesion can be obtained between perfectly smooth surfaces, which shows that although mechanical interlock aids adhesion, it is not a universal mechanism that can be applied and explains all cases.

#### 2.3.2. Chemical and physical adsorption theories

In a physical adsorption process, the bond is established by secondary bonds such as van der Waals forces. These are the most important forces in the adsorption process and intervene in virtually all adhesive bonds. These forces are generated when there is large number of molecular chains of the adhesive in close proximity to the adherend surface, something which can only be achieved with good wetting. A schematic representation of physical adsorption is shown in the left part of Figure 2.10.



Figure 2.10. Physical (left) and chemical (right) adsorption between an adhesive and a adherend.

A chemical adsorption process, in contrast, is achieved via the establishment of chemical bonds between the atoms of the adhesive and of the adherend, as illustrated in the right-hand part of Figure 2.10. The chemical bonds can include acid-base interactions, hydrogen bonds and even primary bonds. Primary chemical bonds are extremely strong and contribute greatly to the level of adhesion in some cases. Chemical adsorption bonds can be enhanced by the use of bonding agents and adhesion promoters (such as primers).
## SURFACE PREPARATION

### 3. SURFACE PREPARATION

The aim of surface preparation is ultimately to optimise the adhesion forces between the adherend surface and the adhesive layer and is an indispensable activity in the preparation of bonded joints employed in aerospace applications, where failure cannot be tolerated.

Modern surface preparation processes comprise of a reasonable compromise between surface wettability, adhesive viscosity and substrate roughness. Ideally, the adhesive should spread across the surface, which means that the chosen adhesive should wet the surface of the adherend, but without detriment to the chemical compatibility (at the intermolecular and chemical interactions level) between the adhesive and the surface layer of the adherend. It should also be ensured that there is no "weak layer" between the adhesive and the adherend (shown in Figure 3.1). This type of layer is relatively common and can have various origins such as contaminant films, oxide layers, corrosion, loose particles, dust or even grease, all of each can be commonly found in the substrate materials used in aircraft construction.



Figure 3.1. Representation of a weak layer in the surface of a adherend.

#### **3.1. SURFACE TREATMENT SELECTION**

To select an ideal surface treatment, it is first necessary to understand the nature and the main physical and chemical characteristics of the materials to be bonded. In fact, this is one of the main challenges associated with surface preparation activities, as adhesive bonding can be applied to a vast number of materials, each with its own set of appropriate preparation techniques.

It should also be noted that, more than influencing the immediate strength of the joint, surface treatments play a key role in the durability of adhesive joints. As shown in Figure 3.2, although many surface treatments show similar performance immediately after bonding, this is not the case in the long term, especially if the joint has been subjected to adverse environmental conditions such as those faced by aeronautical structures during service (especially service under high moisture levels due to exposure to inclement weather).



Time immersed in water

Figure 3.2. The effect of surface preparation on the long-term durability of a bonded joint.

#### 3.1.1. Metals

Surface treatments suitable for use with metallic substrates must be able to control the oxide layer on the surface. While some oxides are strongly bound to the base metals (such as those of titanium), other oxides are arranged in layers with low mechanical strength (in steel and non-anodized aluminium). Nonetheless, the nature of the oxides in supplied materials is often not precisely known, so it is advisable to remove them and replace them with a stable and well-controlled oxide. Ideally, a surface preparation procedure should remove all surface layers until the base metal is exposed.

#### 3.1.2. Polymers

Polymers naturally have low surface energy and very dynamic surfaces that can evolve more easily than those of metals. Surface treatments suitable for polymers mostly act on increasing the surface energy. Furthermore, several substances associated to the manufacture processes (such as release agents) and oriented layers can also be found on these surfaces, which greatly influence the interfacial strength of a joint, even if they do not significantly alter the mechanical properties of the base polymer.

#### 3.1.3. Composites

In structural applications, adhesive bonding is almost always the technique of choice for joining fibre-reinforced polymer matrix composites as it does not require drilling holes or performing other modifications to the composite material. However, it is difficult to ensure optimal adhesion to composites since, as described for polymers, the mostly polymeric surfaces of these materials are usually of low surface energy and often covered in powerful release agents, resultant from their manufacturing processes. While it is essential to clean these contaminants, the use of intensive abrasive treatments is strongly advised against, as they can cause severe surface damage and even damage the reinforcement material.

One particular technique used to prepare composites for bonding is the use of a peel-ply layer. The peel ply is an extra ply of composite material, laminated into the component during the manufacturing process. When adhesive bonding is to be carried out, the peel ply is removed, creating a fractured zone that exposes the resin immediately above the first layer of reinforcement. This is an uncontaminated and rough surface, that will facilitate adhesive bonding, although it does not solve the issue of low surface energy of the polymeric resin.

#### **3.2. SURFACE TREATMENT TECHNIQUES**

Surface treatments used in aeronautical applications are usually classified as passive or active treatments. In passive treatments, there is no modification of the chemical nature of the substrate surface. Such treatments clean and remove weak layers from the surface. While these processes make an important contribution to improving adhesion, in many cases this is not enough to avoid adhesive failure and active treatments are required. Still, passive treatments are almost always used as an initial step in the preparation of a surface, exposing the base material which, if necessary, can then be processed using an active treatment.

Active processes can also be used for cleaning and removing weak layers, but their main advantage is that they change the chemical nature of the surface, which is the reason for the name "active". They are almost indispensable for applications where high strength and

durability are required. These treatments can promote the growth of stable oxides with well-defined structures in metals or the formation of polar groups the surface energy in polymers. These processes can be further divided into physical or chemical treatments, depending on how they modify the surface.

#### 3.2.1. Passive processes

#### 3.2.1.1. Cleaning and degreasing

Cleaning and degreasing processes aim primarily at removing contaminants, oil or grease from the substrates, all of which result in very low levels of adhesion. In many non-structural bonding applications, these processes are often sufficient, however, in structural applications such as those found in aeronautical construction, cleaning is often just the first step in a more complex surface treatment process.

Degreasing is the simplest method of cleaning and is crucial for achieving a contaminant free surface, thus increasing its surface free energy and improving adhesive wetting. This process can be implemented via manual cleaning (shown in Figure 3.3), dipping, spraying, steaming or aided by ultrasonic exposure. The cleaning agents used to perform passive surface treatment must remove all soluble contaminants from the substrate surface and can include solvents, degreasing agents and detergents. Polar agents dissolve polar contaminants, while non-polar agents perform better on surfaces with non-polar contaminants.



Figure 3.3. Manual cleaning and degreasing process using acetone.

In general terms, mechanical, chemical, electrochemical, and some other alternative treatments are known to result in much higher levels of adhesion and durability than those resulting from a simple degreasing process.

#### 3.2.1.2. Abrasive processes

Abrasive surface treatments can be carried out manually or assisted with powered equipment and machinery. These are widely used to prepare the surface of a substrate, representing an inexpensive and practical methodology that does not require highly specific equipment.

Manual abrasion processes, using sandpaper, brushes and metal wool, depend largely on the experience of the operator. If performed incorrectly, they provide inconsistent results and should only be used if no other method is possible. Furthermore, when a dry abrasion process is employed, it is strongly recommended to include an initial degreasing step in the full procedure, preventing surface residues from becoming embedded in the surface to be treated or being transferred to other areas of the surface by the sandpaper, reducing the effectiveness of the process.

Since surface abrasion results in well oriented grooves, it should always be carried out in alternating, crosswise (perpendicular to each other) movements to ensure that there is no preferential direction of treatment on the surface. On completion of the process, particles and dust should be thoroughly cleaned from the surface, which can be achieved using an oil-free blast of compressed air, or by repeating a cleaning process using a solvent or a degreasing agent.

Gritblasting or sandblasting is a widely used surface preparation for metal substrates. In this method, compressed air is used to propel abrasive particles against the surface to be treated, allowing rapid removal of contaminants and oxide layers. This method requires an investment in equipment, although the cost of suitable equipment is not very high due to its relatively simple construction, as shown in Figure 3.4. It should be noted that each type of material requires a specific procedure, using different shapes, particle sizes and operating pressures.



Sandblasting process

Figure 3.4. Sandblasting process of metallic substrates.

In many metals, this type of surface treatment can lead to a noticeable and important increase in the level of adhesion. However, excessive roughness can cause a reduction in resistance due to the formation of pores on the surface, which can be the source of cracks in service, especially in components that are subject to fatigue loads in service.

Furthermore, by removing a layer on the surface of the material, gritblasting and sandblasting can significantly alter the surface stresses present in a metallic component. Given that most metallic components used in aeronautical construction are quite thin, this can have the undesirable effect of creating imbalances in the surfaces, leading to bending and deformation of the components. Thus, the use of these treatments in thin materials should be carried out with extreme caution.

#### 3.2.2. Active processes

Active chemical processes are quite diverse in nature and formulated in a way that is highly optimised towards a well-defined target material. Treatments applied to metals differ greatly from those applied to polymers and polymer matrix composites. In the case of metals, oxide layers and other weak layers are removed through a chemical process, promoting an increase in roughness and leading to an improvement in mechanical adhesion. This follows a course of action that is generally close to that achieved with passive methods. However, in polymers and polymer matrix composites, most chemical treatments seek to activate a surface and create reactive sites, supporting the establishment of van der Waals, chemical and hydrogen bridging bonds. Such treatments can also have a positive (but limited) contribution to the level of mechanical interlocking

#### 3.2.2.1. Active surface treatment of metals

The most basic chemical treatment methods are based on the principle of solubility of the oxides, hydroxides and other surface compounds in the stripping agent. The stripping agent can be an acid ( $H_2SO_4$ , HCl, HNO<sub>3</sub> or HF) or a base (NaOH), and all reaction products are generally soluble in an aqueous medium.

Electrochemical treatments are especially used in the treatment of aluminium and titanium alloys in the aeronautical industry, in a process commonly known as anodising. In this treatment, there is the formation and growth of an oxide layer with a well-defined cellular structure. The treatment takes place in acid filled and electrically charged anodizing baths, where the substrate to be treated acts as an anode (is positively charged). Substrates treated with electrochemical methods are known to exhibit good levels of adhesion, long-term stability and corrosion resistance. The differences between a treated and untreated aluminium alloy surface are depicted in Figure 3.5, as well as the oxide layer formed on the

surface of a metal, showing the pores that can enhance mechanical interlocking with the adhesive.



Figure 3.5. Anodization of aluminium surfaces and schematic representation of a structure formed after an anodization process.

The changes induced by anodization on the surface of metals are especially obvious when observed with an electron microscope. In electrochemical treatments, only ion-conductive oxides (e.g. Al,O3, TiO,) can be deposited in thick layers by anodising,

Special coating baths are also commonly employed in the aerospace sector to prepare the surface of metals for bonding. Historically, chromate conversion coatings have been used in airframe components, serving both as corrosion inhibitors and as primers to improve the adhesion of the metal to paints and adhesives. These coatings are also known to provide resistance to abrasion and to light chemical attack. However, the presence of hexavalent chromium compounds in their formulation, known to be highly carcinogenic, has led to their replacement in many applications with alternative formulations that do not contain this chemical.

An important alternative to chromate conversion coating are the sol-gel coatings. Not unlike the former, these are mainly used on metallic substrates to improve chemical stability and adhesion. A sol-gel coating entails the formation of a thin, hard and homogeneous ceramic coating. The sol-gel precursors are supplied on an aqueous form, called sol, that will be deposited on the surface of the substrate. This liquid includes alkoxides and other compounds that will undergo first and hydrolysis reaction, followed by a condensation reaction.

In many cases, aluminium components used in aircraft construction are supplied in the Alclad state, which uses a very thin layer of pure aluminium metallurgically bonded to a

high strength aluminium alloy core. While the Alclad sheets are quite durable and resistant to corrosion, it is also advisable to prepare the areas to be bonded with a surface treatment which promotes an increased level of adhesion, such as a localized anodization process or a sol-gel coating.

#### 3.2.2.2. Active surface treatment for polymers and polymer matrix composites

A wide range of active treatments are suitable for the preparation of polymeric and polymer matrix composite substrates. However, flame, plasma and corona discharge treatments are those which have more relevance for bonding composites and polymers in the aerospace sector and thus are described in more detail in the following paragraphs.

#### Flame

Flame treatment is a very simple and inexpensive surface treatment for polymers that is carried out via the exposure of the surface of substrates to a flame. The aim of this treatment is mainly oxidation of the polymer surface, leading to the formation of chemical complexes which are favourable to the establishment of the desired bonds. The phenomena intervening at the surface are of the same type as those occurring in alternate methods such as plasma treatment or corona discharge. However, this is a more energy-dense method, which has the potential to damage substrates if used incorrectly. Its effects deteriorate rapidly over time as the treated material, now made to be highly active, will quickly chemically react with the atmosphere. Consequently, bonding of flame treated substrates should be carried out immediately after treatment or within a very short period of time.

#### Atmospheric and low-pressure plasma

In a plasma treatment, polymers are exposed to a highly energetic jet of plasma, capable of changing their surface chemistry. The plasma consists of a mass of air that has been subjected to a strong electrical discharge, which causes the dissociation of the molecular bonds of its constituent gases, transforming it into a plasma containing charged particles (mainly electrons and positive ions) that react easily with polymeric materials. Most polymeric materials have chemically inert, non-porous surfaces with low surface energy, which are not very receptive to adhesive bonding, but the use of plasma forms a chemically active surface that readily adheres to adhesives. Plasma treatment can be carried out with ionised air (atmospheric plasma) or using a specific gas, ionised inside a sealed and pressure-controlled chamber (low pressure plasma), although this greatly restricts the size of the component that can be treated and is thus of more limited industrial importance. An example of an atmospheric plasma treatment of a composite material and its effect on the contact angle is shown in Figure 3.6.



Figure 3.6. Plasma treatment procedure on a composite substrate and its effect on wetting.

#### Corona discharge

Corona discharge treatment (CDT) is a surface modification technique that also relies in a plasma to promote changes in the properties of a surface. A corona discharge plasma is simply highly ionised air surrounding a highly electrically charged conductive material that is exposed to the atmosphere. This treatment was initially developed to treat polymeric films intended for painting and coating processes. While atmospheric and low-pressure plasma is preferred for structural applications in the aerospace sector, corona discharge treatments still find use in the treatment of simpler shapes, such as tubing and cabling, providing faster processing rates.

# ADHESIVES USED IN AEROSPACE APPLICATIONS

### 4. ADHESIVES USED IN AEROSPACE APPLICATIONS

This chapter reports the main types of adhesives used in aerospace applications, describing their advantages, limitations and typical uses. As stated in the introductory section, these adhesives are typically classified into structural and sealants, which serves to highlight the role they have in an aeronautical structure.

One important note is the fact that although one might reference the adhesive itself as a single product, nowadays it is common to see adhesive supplied as part of so-called systems. In this case, the system represents all the materials necessary to carry out the bonding process and, besides the adhesive, might also include suitable coating, primers or chemicals for surface treatment. Such systems allow the manufacturer of the adhesive to create a closed set of conditions that have been tested and validated and this solution allows for increased control of the process and better assurances of joint performance.

#### 4.1. STRUCTURAL ADHESIVES

#### 4.1.1. Epoxy adhesives

Epoxy adhesives, informally known as the kings of adhesives, are strong and rigid materials with an extensive history of use in structural applications (Figure 4.1). In structural applications where maximum mechanical performance is indispensable, epoxy adhesives are often the only viable choice.



Figure 4.1. Summary of the key characteristics of epoxy adhesives.

Epoxy adhesives can be supplied as a separate resin and hardener (two parts) or as a single component and cure exothermically (i.e. they release heat during the hardening process). These are very dimensionally stable materials, with a relatively low level of shrinkage. Shrinkage is the reduction in volume that the adhesive undergoes during curing, something that is detrimental to the geometric characteristics of the joint, can cause warping when thin substrates are used and that can also induce undesirable stresses in the adhesive layer. In general, cured epoxies are very stable and durable, and are not damaged by contact with water, solvents or oils.

Single component epoxies only harden on exposure to high temperatures. However, they cannot be stored for long periods of time, especially if they are not kept under refrigerated conditions. Two-part epoxies, on the other hand, cure at room temperature and can be stored for longer periods at room temperature.

As highly cross-linked thermosets, epoxies are known to be strong and rigid, but rather brittle. Hence, commercial versions of these materials are rarely pure, being in fact hybrid formulations, reinforced with small amounts of rubber or other tough polymers. In unreinforced and brittle epoxies, cracks can progress quickly and unhindered, but the presence of small rubber particles will prevent the free propagation of cracks. It is also quite common to find epoxy adhesives reinforced with other materials, targeting an improvement of a specific property. For example, epoxy-polyurethane hybrids are extremely popular in the automotive industry, combining the stiffness and strength of epoxy resin with the toughness of polyurethane, which gives them excellent impact absorption capabilities. The aeronautical industry makes a limited use of impact resistant formulations, applying them in the construction of engine nacelles which must contain the engine failures and sections of

the leading edges that are susceptible to damage due to bird-strikes. More commonly used are alternative epoxy formulations which enhance temperature resistance while retaining excellent mechanical strength. The characteristics of some important commercial epoxy adhesives used in aerospace applications are summarized in Table 4.1. below.

| Commercial<br>reference | Manufacturer         | Form                                | Cure conditions                             | Service<br>temperature<br>(°C) | Key properties  |
|-------------------------|----------------------|-------------------------------------|---|--------------------------------|---|
| FM73                    | Solvay               | Film (supported<br>and unsupported) | 1h @ 120°C, plus<br>0.48 MPa of<br>pressure | 82                             | Good peel resistance. Good<br>resistance to moisture and<br>corrosion.  |
| FM300                   | Solvay               | Film (supported<br>and unsupported) | 1h @ 175°C, in<br>autoclave                 | 150                            | Compatible with many materials.<br>High shear strength. Good<br>resistance to moisture and<br>corrosion.                        |
| Redux 810               | Hexcel<br>Composites | Two-part paste                      | 5 days @ 22°C or<br>1h @ 70°C               | 100                            | Easy to mix. Good resistance to peel, shear and corrosion.  |
| Redux 322               | Hexcel<br>Composites | Film (supported and unsupported)    | 1h @ 175°C, in<br>autoclave                 | 200                            | Good resistance to very high temperatures. Good shear strength.   |
| AF 163-2                | 3M Scotchweld        | Film (supported<br>and unsupported) | 1h30 @ 120°C                                | 121                            | Excellent performance on<br>composite substrates.<br>Good moisture resistance.<br>High fracture toughness and peel<br>strength. |

 Table 4.1. Characteristics of some epoxy adhesives used in aerospace applications.

#### 4.1.2. Phenolic adhesives

Phenolic adhesives (Figure 4.2) are relatively inexpensive adhesives that are usually supplied in liquid or film form and were the first synthetic adhesives specifically formulated for aeronautical construction. The Redux (REsearch at DUXford) phenolic-based adhesives were first formulated in 1941 by Aero Research Limited, for bonding of metal-to-metal and metal-to-wood structures and found use in many British built aircraft of World War II. In fact, many Redux adhesives are still used today. For example, the Redux 775 film adhesive, developed in 1962 is still found in diverse applications in the aircraft industry, since it has been extensively validated in service and thus represents a reassuring proposition for the naturally risk-averse aeronautical designers.



Figure 4.2. Summary of the key characteristics of phenolic adhesives.

Phenolic adhesives are thermosetting resins (do not melt with temperature) that can withstand high temperatures (up to 180°C), harsh environmental conditions (high humidity and exposure to oil and solvents), are fire resistant and exhibit low creep. However, they are relatively brittle and exhibit low resistance to peel.

A major challenge associated to the use of phenolic structural adhesives is their curing process, which requires simultaneous application of heat and pressure. Since these adhesives harden through a condensation process (as shown in Figure 4.3), the presence of free hydrogen and oxygen leads to formation and release of water molecules in the adhesive during hardening. Since this occurs at high temperature (usually over 100°C), vapour bubbles are formed and trapped in the cured adhesive, creating a highly porous adhesive layer, something that can be avoided by using a curing process carried out under constant pressure, necessarily larger than the vapour pressure of water.



Figure 4.3. The condensation curing process, showing the release of water molecules.

As is the case for the other adhesives presented in this section, phenolics can also be supplied as modified adhesives. The most common among these are nitrile-phenolic, vinyl-phenolic

and neoprene-phenolic adhesives. They are supplied as solvent solutions and as supported and unsupported films. In the case of solvent-based adhesives, evaporative hardening is used. A summary of some phenolic adhesives used in aerospace applications is provided in Table 4.2.

| Commercial<br>reference | Manufacturer         | Form                         | Cure conditions                              | Service<br>temperature<br>(°C) | Key properties   |  |  |
|-------------------------|----------------------|------------------------------|--|--------------------------------|--|--|--|
| Nitrile-phenolics       |                      |                              |  |                                |  |  |  |
| L 728                   | Solvay               | Resin                        | 1h @ 135°C, in<br>autoclave or vacuum<br>bag | 82                             | Airtight for use in ducts. Suitable<br>for reinforcement with fibres. Fire<br>resistance.  |  |  |
| PL 663                  | Loctite              | Film                         | 135 min @ 152°C, pus<br>0.69 MPa of pressure | 132                            | Good shear strength. Good resistance to high temperatures.   |  |  |
| 583                     | зM                   | Film                         | 20 seconds @ 150°C                           | 149                            | Good resistance to fuels and other<br>chemicals. Highly flexible and good<br>peel strength. Good resistance to<br>high temperatures. |  |  |
| Vinyl-phenolics         |                      |                              |  |                                |  |  |  |
| Redux 775               | Hexcel<br>Composites | Film or liquid<br>and powder | 30min @ 150°C, plus<br>0.7 MPa of pressure   | 70                             | First aircraft rated adhesive. Good<br>environmental and corrosion<br>resistance.  |  |  |

 Table 4.2. Characteristics of a few phenolic based adhesives used in aerospace applications.

#### 4.1.3. Aromatic adhesives

Aromatic adhesives (Figure 4.4) are by far the most temperature-resistant structural adhesives, capable of operating at maximum temperatures above 280°C, almost 100°C higher than all other structural adhesives. They find use almost exclusively in aerospace applications, for example in bonding the airframes in supersonic aircraft which, due to intense frictional heating, can face temperatures over 230 °C for extended periods of time (over 3 hours).



Figure 4.4. Summary of the key characteristics of aromatic adhesives.

Almost exclusively supplied in film form, they are all very costly materials, difficult to process and specifically formulated for bonding components that will be subjected to intense temperatures. Although extremely resistant to aggressive environments, they are quite limited in their mechanical performance, being brittle and with low resistance to peel loadings. Moreover, their toughness cannot be increased by the addition of other materials, as is the case with other adhesives.

Two main types of aromatic adhesives, polyimides and polybismaleimides, can be found on the market. Polyimides, provide excellent thermal resistance which is superior to any other organic adhesive. These modified polyimides are single component, solvent-based adhesives which can operate at temperature above 240°C. Polybismaleimides (BMIs) are high performance thermosetting polyimides which cure by addition. Their characteristics are generally similar to that of polyimides, exhibiting high strength and stiffness at extreme temperature temperatures, long-term stability to chemical degradation, good electrical properties and relatively low moisture absorption. Examples of aromatic adhesives commonly used in aerospace applications are given in Table 4.3.

| Commercial<br>reference | Manufacturer      | Form                               | Cure conditions  | Service<br>temperature (°C) | Key properties  |
|-------------------------|-------------------|------------------------------------|--|-----------------------------|---|
|                         |                   | Polybism                           | naleimides   |                             |   |
| Metlbond 2550           | Solvay            | Film with support                  | 6h @ 177ºC under<br>pressure. Post-cure<br>6h @ 227ºC                  | 204                         | High compressive<br>strength. Resistant<br>to solvents.<br>Outstanding bond<br>strength with<br>composites and<br>Metals. |
| Redux HP655             | Hexcel Composites | Film (with and<br>without support) | 4h @ 191ºC, plus<br>0.4 MPa. Post-cure<br>16h @ 232ºC                  | 240                         | High toughness and shear strength.  |
| Hysol EA 9673           | Henkel            | Film                               | 1h @ 177°C under<br>pressure. Post cure<br>of 2h @ 246°C               | 288                         | Good resistance<br>to very high<br>temperatures.<br>Moisture resistant.   |
|                         |                   | Polyi                              | mides  |                             |   |
| FM 57                   | Solvay            | Supported film                     | 90min @ 177°C,<br>plus 0.2 MPa of<br>pressure. Post-cure<br>2h @ 288°C | 288                         | Compatible with<br>a wide range of<br>materials. Relatively<br>short processing<br>times.                                 |
| FM 680                  | Solvay            | Film                               | 2h @ 177°C at 0.69<br>MPa of pressure.<br>Post-cure 2h @<br>271°C      | 371                         | Extreme thermal<br>resistance and<br>stability. Low<br>oxidation.   |

| Table ⊿.3. Characterist | ics of a few aron | natic adhesives used | l in aerospace applications. |
|-------------------------|-------------------|----------------------|------------------------------|
|                         |                   |                      | in der eepace appricationer  |

### 4.1.4. Comparison between the performance of different structural adhesives used in aeronautical construction

Figure 4.5 shows a comparative analysis of the strength and the service temperature of different structural adhesives used in aeronautical construction. This comparison clearly shows that phenolic adhesives are generally weaker than epoxy and aromatic adhesives and can be used up to 200 °C. One component epoxies are the strongest of these group, possessing extraordinary strength at room temperature. However, their performance drops quickly as the temperature rises. Finally, the aromatic adhesives can retain their strength at very high temperature, offering unrivalled performance under these extreme conditions.



Figure 4.5. Comparison of the strength and service temperature of different adhesives used in the aeronautical industry.

#### 4.2. NON-STRUCTURAL ADHESIVES AND SEALANTS

#### 4.2.1. Elastomeric adhesives

Elastomeric adhesives (Figure 4.6) are very flexible materials and can be based on natural or synthetic rubber-based materials. These adhesives do have excellent peel strength and toughness but exhibit low shear strength, which makes them unsuitable for most structural bonding applications. Still, their extreme elasticity and toughness results in good fatigue and

impact performance and elastomeric adhesives are found in applications where the joint is subjected to large movements, impact or any other conditions where there is significant relative movement of the bonded adherends. This includes, for example, the displacements caused by thermal expansion of dissimilar materials. Being relatively inexpensive, impervious to water ingress and exhibiting good adhesion to most aeronautical construction materials, these adhesives are also widely used as sealants in primary aeronautical structures.



Figure 4.6. Summary of the key characteristics of elastomeric adhesives and sealants.

Room temperature vulcanizing (RTV) silicones are a special type of elastomeric adhesive which exhibits extremely high temperature resistance. This allows them to be used in extreme applications such as the thermal protection systems for aerospace applications, such as those discussed in Chapter 1. Still, most elastomeric adhesives react poorly to high temperatures and cannot be used above 100°C.

Another important disadvantage associated with these adhesives is their susceptibility to creep deformation. Creep is the slow and gradual adhesive displacement that occurs under relatively low mechanical loads, lower than those necessary to break the adhesive under quasi-static conditions. If creep is left unchecked for long periods of time, an adhesive joint can eventual fail due to excessive deformation. Chapter 9 provides more detail on the creep phenomenon.

For most adhesives, creep failure is only a factor at high temperature, but for elastomeric adhesives it can occur at room temperature. This means that a relatively unloaded joint held together by these adhesives can easily deform if not properly designed to account for creep. In aerostructures, this issue is often solved by relying on riveting to hold the adherends in place, while a silicone adhesive is relegated solely to the role of a sealant, not transferring any loads.

#### 4.2.2. Inorganic adhesives

Although the overwhelming majority of modern adhesives are based on polymeric formulations, adhesives based on inorganic compounds also exist (Figure 4.7). The most common inorganic adhesives have a ceramic or metallic base and are extremely resistant to high temperatures, in some cases capable of operating at over 600°C. Although they can be roughly compared to the concrete used in civil engineering, ceramic adhesives are based on alkali silicates and various metal phosphates, supplied in powder form.



Figure 4.7. Summary of the key characteristics of inorganic adhesives.

During the hardening process, a dehydration and chemical reaction process occurs, assisted by a binder, creating a solid and refractory layer. It should be noted that the ability to withstand high temperatures exhibited by these materials has some important drawbacks, such as their high cost and complex curing process. The curing temperature depends on the binder used and can range from a minimum of 250°C, up to 1000°C, far above the temperatures used to cure organic adhesives and necessitating very different equipment.

However, the most important limitation associated to the use of these adhesives is the fact that they are extremely fragile and also have a very low resistance to peel and shear. Some products available on the market bypass these limitations by featuring a hybrid formulation, using a high temperature epoxy adhesive filled with ceramic or metallic powders. These adhesives can offer the high bond strength, low shrinkage and machinability of epoxy adhesives, combined with the high temperature stability, thermal shock resistance and chemical resistance of inorganic adhesives and find use in combustion engines, rocket engines and gas turbines, especially for potting and encapsulating electronics.

#### 4.3. MECHANICAL CHARACTERIZATION OF ADHESIVES

The mechanical characterisation of adhesives can be performed through a wide variety of tests, allowing to determine parameters such as strength, stiffness and toughness. Using these tests, it becomes possible to distinguish rigid, strong and brittle adhesives from those that are more ductile, flexible, and capable of absorbing energy, serving as an indispensable support for the adhesive selection processes. Figure 4.8 shows how a simple tensile test can be used to identify the different nature of two adhesives.



Figure 4.8. Comparison of the tensile performance of two highly distinct epoxy-based adhesives.

#### 4.3.1.1. Bulk tensile and compression testing

Tensile tests allow to determine how an adhesive (and not the bonded joint) will behave when loaded under tension. In this case, a sample of the material will be tensioned and will try to resist this load until its eventual rupture occurs. By collecting load and strain information during this test (using load cells and a strain gage system) it becomes possible to create a stress-strain curve and thus accurately characterise the stiffness, strength and yield stress of an adhesive subjected to tensile loads. An example of a resultant stress curve is shown in Figure 4.9.



Figure 4.9. Main mechanical properties that can be extracted from a tensile test.

Tensile testing of adhesives can also be used to obtain an estimate of the total deformation and ductility of the material (its ability to deform before breaking). However, bulk tensile testing specimens are not ideal for this purpose since this measurement is highly dependent on defects in the adhesive.

It is also possible to subject adhesives to compression tests, which require the use of a system to stabilise the adhesive specimen to prevent buckling failure. Although the stiffness of an adhesive under compression is very similar to the one obtained in tension, compressive strengths are usually 20% to 40% higher. Nonetheless, adhesives are rarely used to support compression loads in aerospace applications, since there are many other joining processes that can do so effectively and in a simpler manner.

#### 4.3.1.2. Shear testing

Shear tests of adhesives (or mode II tests) require two parallel but opposite loads that produce a shearing effect on the material. Shear tests are arguably more relevant for testing adhesives that will be used in structural applications than tensile tests, since a shear condition distributes the load more evenly throughout the adhesive layer, creating much lower peel loads and are often more representative of the loads adhesives will face in service. There are several test methodologies suitable for performing shear tests, each with different characteristics. The simplest of these is perhaps the Thick Adherend Shear Test (TAST). This test uses a geometry that closely resembles a single overlap joint but using thick adherends, a feature which ensures minimal joint rotation during the test. This almost completely eliminates peel loads, ensuring an almost pure shear state in the adhesive layer. The geometry of a TAST specimen is shown in Figure 4.10



Figure 4.10. Geometry of the TAST specimen (left, dimensions in mm). Example of a tested specimen (right).

#### 4.3.1.3. Fracture toughness testing

When a crack grows in a material, it generates new surfaces, exposing atoms that have a higher potential energy than those that are not disturbed within the material. A crack will only propagate if the reduction in potential energy is greater than the energy required to create the new crack surface. The critical energy release rate, or the fracture energy, corresponds to the minimum energy that must be supplied to the material to initiate crack propagation. Using an adhesive with a high fracture energy will ensure that the bonded joint is able to resist more damage before it fails, leading to a joint that also fails in a much safer and predictable way, something which is highly desirable in the aerospace sector.

A commonly used method for the determination of the fracture energy of adhesives under tensile load (mode I) is the Double Cantilever Beam (DCB) test (Figure 4.11). In this test, two parallel adherends are joined by a thin adhesive layer. The specimen is then subjected to a tensile load at one of its ends, creating a crack that propagates parallel to the adherends, along the adhesive layer.



Figure 4.11. Examples of DCB specimens and its key features.

Initially, fracture energy determination could only be achieved with a direct measurement of the crack location as the test progresses, accomplished by loading and unloading the specimen repeatedly and registering the load and the crack location for each of these cycles, or by controlling the crack location with an optical measurement method.

The Compliance Calibration Method (CCM) is perhaps the simplest method that can be used to calculate the critical fracture energy. This method is based on the use of the Irwin Kies equation.

The determination critical fracture energy starts with the definition of the total potential energy of the system ( $\Pi$ ). This energy is the difference between the elastic energy stored by the body (U) and the potential energy of the external force applied to body. The potential energy is of the external force is obtained by multiplying the displacement ( $\delta$ ) and the load (P). This is shown in Equation 4.1 and this relationship is schematically represented in Figure 4.12.

$$\Pi = U - P\delta$$
 4.1



Figure 4.12. Relationship between the total potential energy of the system ( $\Pi$ ) and the elastic energy stored by the body (U).

Based on what is shown in Figure 4.12, we can write the total potential energy of the system as laid out in Equation 4.2.

$$\Pi = -\frac{1}{2}P\delta$$
 **4.2**

Additionally, the compliance of the specimen being tested (*C*) is obtained by simply relating the displacement and the load acting on the specimen (Equation 4.3).

$$\delta = CP \qquad \qquad \textbf{4.3}$$

However, the compliance will evidently be dependent on the crack length (*a*). The longer the crack in the specimen, the more it will flex for a given load. Classical beam theory, first developed by Euler and Bernoulli, can be used to quantify this relationship. Using a simplification of the linear theory of elasticity provides a relatively accurate mean of calculating the deflection characteristics of a beam, as a function of its properties and the applied load.

The beam theory can be used to define the displacement as a function of the crack length, the load applied to the specimen, the stiffness (*E*) and the moment of inertia (*I*). The moment of inertia is a purely geometrical property that measures the ability of a beam to resist bending. This allows rewriting Equation 4.3 solely as a function of geometrical parameters such as the width (*b*) and the thickness (*h*) of the beams that compose the DCB specimen (Equation 4.4).

$$\delta = 2 \frac{Pa^3}{3El} = P \frac{8a^3}{Ebh^3}$$

The compliance can be defined as a function of the crack length, (Equation 4.5).

$$C(a) = \frac{8a^3}{EBh^3}$$
**4.5**

Taking this into account, the total potential energy in the system can be written simply as a function of the load acting on the specimen, the crack length and the dimensions of the specimen (Equation 4.6)

$$\Pi = -\frac{1}{2}P^2C(a) = -\frac{1}{2}P^2\frac{8a^3}{Ebh^3}$$
**4.6**

As the potential energy equation can be used to determine the fracture energy, we finally obtain the Irwin Kies equation by considering the variation of the potential energy ( $d \Pi$ ) per unit of area (*bda*). This is Equation 4.7, which is suitable for the calculation of the critical fracture energy (*G*).

$$G = -\frac{d\Pi}{bda} = \frac{P^2}{2b} \frac{dC}{da} = 12 \left(\frac{P}{bh}\right)^2 \frac{a^2}{Eh}$$
**4.7**

Simply put, to determine the critical fracture energy in a DCB specimen it is necessary to obtain the load and displacement curve of the test, provide the specimen width and control the evolution of the crack length during the test.

Other methods are also available to process the DCB testing data, but they all rely on a visual (and subjective) identification of the crack location. This has led to the emergence of novel methodologies which do not require direct measurement of the crack location as they can provide an accurate estimate of its location based only *P*- $\delta$  data. One of the most important of these is the Compliance Based Beam Method (CBBM), that considers the deflection of the DCB specimens and uses this data to determine the effective location of the crack tip. More precisely, CBBM assesses the compliance of the complete DCB specimen and uses this information to determine the crack length (using an approach which is also based on the classical beam equations) This method also accounts for the damaged area immediately ahead of the crack tip, known as the fracture process zone, which cannot be visually detected.

#### 4.4. EFFECT OF TEMPERATURE ON ADHESIVE PROPERTIES

As shown schematically in Figure 4.13, the mechanical properties of an adhesive (such as its stiffness and strength) strongly depend on the service temperature or, more precisely its proximity to the glass transition temperature ( $T_g$ ). The  $T_g$  is a temperature at which the adhesive response changes from strong, stiff and brittle to a rubber-like behaviour. For service temperatures well below  $T_g$ , the modulus and strength of the adhesive rise significantly to the detriment of its ductility. In contrast, as the service temperature increases towards  $T_g$  there is a rapid reduction in stiffness and strength. Above  $T_g$ , a structural adhesive will no longer be able to withstand substantial loads, exhibiting a behaviour similar to that of a sealant at room temperature.



Figure 4.13. Schematic representation of the influence of temperature on the tensile behaviour of a structural adhesive.

This has very important implications for aerospace applications, where adhesives and sealants are often exposed to service under extreme thermal conditions, as is the case for panels subjected to supersonic airflow, engine components and thermal protection systems in spacecraft. Accordingly, precise determination of the T<sub>g</sub> of an adhesive is an indispensable step to assure a durable bonded structure which performs well within the expected service range. A more detailed analysis of the effect of temperature on the durability of adhesives and bonded joints is made in Chapter 9 of this book.

#### 4.5. EFFECT OF MOISTURE ON ADHESIVE PROPERTIES

The presence of moisture has a very substantial effect on the performance of a structural adhesive. Subjected to a moist environment, both the adhesive and the adhesive-adherend interface may absorb water molecules. These molecules can form bonds with the polymeric chains of the adhesive or occupy free spaces within the adhesive until a saturation situation is reached. The adhesive will become less rigid and generally weaker as the absorbed water content increases. However, the ductility of the adhesive does tend to increase since the presence of water forces the polymer chains to expand, facilitating its relative movement. A summary of the effect of moisture on adhesives behaviour is clearly shown in Figure 4.14.



Figure 4.14. Schematic representation of the influence of moisture on the tensile behaviour of a structural adhesive.

As reported for the effect of temperature, the presence of moisture should also be an important concern for designers of aeronautical bonded structures. During service, an aircraft will face extreme moisture levels and direct contact with water. The adhesive joints in an airframe will almost certainly absorb some of this moisture and it is crucial to ensure that the degradation in adhesives properties is known and accounted for in the design phase. Further details on the effect of moisture on the long-term durability of adhesives and bonded joints can be found in Chapter 9.

#### 4.6. EFFECT OF STRAIN RATE ON ADHESIVE PROPERTIES

The strain rate, i.e. the speed at which an adhesive or a joint is deformed, also has an important effect on the mechanical behaviour of an adhesive. Adhesives, like most polymers, exhibit a viscoelastic and viscoplastic behaviour and therefore have mechanical properties that are strongly time dependent. Typically, an adhesive under impact conditions will exhibit increased stiffness and strength, as shown in Figure 4.15. Paralleling the effect of low temperatures, it is also usual to encounter a reduction in ductility associated to this condition.

The response of an adhesive to strain rate is mainly caused by the relative movements of the polymeric chains. For low rates, the chains have time to easily slip over each other and this results in low strength and large displacements. But at high rates, the polymeric chains are loaded so fast that time for movement is limited. In practice, they become locked in place, which results in a significant increase in stiffness and strength.





The issue of strain rate sensitivity of an adhesive is admittedly less important for aeronautical applications than the response to moisture and temperature since aircraft structures are rarely designed to sustain impacts. Nonetheless, aerospace applications exist that can benefit from the additional resistance to impacts conferred by the time-dependent behaviour of adhesives. These include, for example, the thermal protection heatshields in spacecraft, which must remain bonded in place, providing protection after important impact loads, nacelles in the nosecones of aircraft and impact absorbing helicopter seats.

## JOINT MANUFACTURING

### 5. JOINT MANUFACTURING

Until this point, we have seen that the use of adhesives in aerospace applications and construction has several important advantages and that many materials exist that fulfil the highly specific requirements of this sector. However, all these benefits would be moot if the actual bonding process could not be carried out in an efficient and effective manner. It is often said that the design of the aerostructure is not the most difficult step and that the real challenges appear once one starts to build and manufacture the real vehicle or component. The same is also generally true for the bonded joints, since a well thought design process can be easily thwarted by a flawed manufacturing process, which does not properly consider the specificities of the adhesive bonding process.

#### **5.1. MANUFACTURE PROCESS**

Regardless of the type of the structure where adhesives are used, a joining process follows all, or many of the specific steps shown in Figure 5.1. Among these manufacturing steps, the aerospace industry generally accepts that surface preparation is the most critical part of the manufacturing process since an error in surface treatment can significantly increase the risk of interfacial failure and subsequently can drastically reduce the joint strength. However, in a qualified manufacturing process, all of the manufacturing stages shown in Figure 5.1 should be correctly performed. In this section the procedure and requirements of the manufacturing of adhesive joints are presented and briefly discussed.



Figure 5.1. A typical adhesive joint manufacture process.

#### 5.2. ADHESIVE STORAGE

Most adhesives are reactive compounds that can degrade easily if the storage conditions are not respected. Storage ageing is an important phenomenon that can significantly degrade adhesive properties and is closely associated to an exponential increase in the viscosity of the adhesive. The maximum allowable storage period and its conditions are usually recommended by the manufacturer in the technical data sheets associated to a given adhesive system. For example, an elevated storage temperature for a film adhesive (which is commonly used in aeronautical industries), can reduce the shear strength by 20%. Moisture has a more damaging effect, eventually leading to significant drops in shear strength, to the order of 40%. Shelf-life (an indication of the maximum time that an adhesive may be stored), and ambient conditions including temperature, humidity, light and UV radiation, and contamination are the most important parameters that should be monitored during adhesive storage. For one component, high temperature curing adhesives, the curing reaction is always slowly progressing even at room temperature. Moisture can be harmful to the adhesives through two mechanisms: ageing (by moisture absorption) and hardening (for those adhesives where moisture acts as a catalyst for polymerisation).

When adhesives are stored in refrigerated conditions, they must be gradually warmed up to the application temperature (room temperature or slightly above to facilitate application). During this process, the containers should remain closed since water will condense on the initially cool container surface. If the container is damaged or not properly sealed, this moisture will condense on the adhesive and can degrade its properties.
# **5.3. SURFACE PREPARATION**

As we have discussed in more detail in Chapter 3, the surface treatment is an essential part of any joining process, playing a key role in the establishment of effective adhesion between the adhesive and the adherend. Depending on the material of the adherend and the type of adhesive, different surface treatments are considered in aeronautical sectors. Anodization, sand blasting, chromic acid etching, chromic acid anodizing, and plasma treatment are some common techniques used to prepare the adherends' surfaces. Using primers at the interface is also a common technique in aircraft industries to reach a good adhesion between the adhesive and the adherends. Primers can not only improve interfacial strength but can also extend durability of the treated surface of the adherend, allowing for longer wait periods during the manufacturing processes.

# 5.4. ADHESIVE METERING, MIXING, AND APPLICATION

#### 5.4.1. Paste and liquid adhesives

As we have seen, many adhesives found in aerospace applications are supplied as two separate components. An incorrect ratio of the resin and the hardener for the two-part adhesives leads to a product with vastly different properties, requiring a precise control of the volume (or mass) of the two components. After metering, the two parts should be mixed together. In small-scale operations, the two parts (resin and hardener) can be mixed manually but for large scale production automated processes are usually employed, using powered mixing machines.

Regardless of the mixing approach used, the main concern during this process should be to ensure that voids are not introduced in the mixture. The air entrapped in these voids can significantly reduce joint strength by creating severe stress concentration points. Voids can also speed up the ageing process (creating more pathways for moisture absorption) and also reduce the effective bonding areas. The presence of voids is of course more damaging in brittle adhesives and in designs where the joints are subjected to tensile and peel loads. A common technique used for removing voids out of the adhesive is the use of vacuum chambers or vacuum bags, enabling the adhesive mixture to be kept at low air pressure (near zero).

After mixing, the adhesive should be applied to the surface of the adherend. The dispensing process can be fully manual for small bonding areas as in, for example, the bonded repair of a damaged airframe panel. Alternatively, it can be performed robotically for larger areas, such as those found, for example, in a vertical stabilizer. The pot life and viscosity of a paste adhesive also determine if the manual application or a robotic process should be employed.

The pot life is the time that the adhesive can be applied after the mixing process since hardening will progress quite swiftly.

Regardless of the method used for adhesive dispensing, the adhesive bead shape should be precisely controlled during the adhesive application. Figure 5.2 shows examples of good and incorrect bead shapes. In the upper part, we can see bead shapes with are likely to trap air, since they provide little or known routes for air escape. The lower part of the Figure 5.2 shows shapes which do not restrict airflow. The wavy bead shape in the lower right corner of special importance, since it provides good area coverage with a single bead.



Figure 5.2. Comparison of paste adhesive application patterns.

The use of liquid adhesives is much more limited in the aerospace sector, since they do not match well with the precise tolerances found in these applications. These adhesives flow very easily out of the gaps and thus have a greater likelihood of introducing defects in the adhesive layer. However, one extensive use of liquid adhesives is in thread locking of fasteners, a safety critical activity that ensures that threaded connections remain in place even under harsh environmental conditions and vibrational loads.

## 5.4.2. Film adhesives

To avoid some of the previously mentioned issues associated to paste and liquid adhesives, adhesives in the form of films are extensively employed in the aeronautical and aerospace industries. The use of films offers some important advantages over paste and liquid adhesives such as the minimum waste, no mixing issues, easier processing, excellent repeatability in adhesive thickness and a more consistent application process, not dependent on the shape of the adhesive beads. However, film adhesives are not a universal solution, since they are not suitable for use in curved or irregularly shaped components. Additionally, film adhesives are generally very expensive, which increases the costs of the manufacturing process and thus tends to limit its use solely to applications where the advantages brought by these materials are indispensable, such as airframes for high performance aircraft which will operate at longer periods of time at elevated temperatures.

Film adhesives are also susceptible to some specific types of defects which arise from their form (Figure 5.3). For example, air can easily be entrapped underneath the layer of film adhesive if the application is not made in a manner which facilitates its release. The presence of voids within the film layer itself can occur mainly for adhesives which cure via a condensation process. In this case, the application of pressure is an indispensable step.



Figure 5.3. Typical defects associated to the application of film adhesives.

The presence of these defects has an important effect on the strength of the film adhesive, and these can have a damaging effect which is more substantial than that found for paste adhesives. This is especially likely for the high temperature applications typical of film adhesives.

# 5.5. JOINT ASSEMBLY

After dispensing or applying the adhesive, the different components of the joint should be assembled and fixed until full hardening of the adhesive is eventually obtained. This operation is known as joint assembly. The main objective of joint assembly is to ensure that the surfaces to be bonded remain in contact with the adhesive, that the dimensions of the adhesive layer are constant and that the adhesive can fully wet the surfaces of the adherends. Furthermore, the application of pressure can also be necessary during this step, being crucial to prevent voids and porosity in adhesives that cure by condensation such as phenolics and polyimides. Controlling the dimensions of the joint, adherend alignment and adhesive thickness is achieved via the use of specialized fixtures. In actual applications in the aeronautical industry such as airframes, complex clamping apparatus are used considered to fix the components to be bonded during the hardening process. Moulds are also extensively used in research and development applications, serving as invaluable tools to reliably manufacture standardized specimen geometries for adhesive and joint performance characterization (Figure 5.4).



Figure 5.4. Example of a mould used to manufacture geometrically precise specimens and components.

However, many of these fixturing devices struggle to control the effective separation between the components to be bonded, leading to incorrect adhesive layer thickness values. Thus, the use of hollow glass beads on the adhesive layer to control the adhesive thickness of the adhesive layer has grown in popularity.

The carriers of film adhesives can also play a similar role, since it represents a relatively incompressible medium that will oppose the direct contact between the two surfaces being joined. This is shown in Figure 5.5.



Figure 5.5. Typical structure of a film adhesive, showing the carrier used to control adhesive thickness.

# 5.6. ADHESIVE HARDENING

The process of hardening adhesives is highly dependent on the nature of the selected adhesive. Adhesives which harden with temperature can be subjected to stages in oven, autoclaves or even in hot-plate hydraulic presses. Autoclaves, large pressurized and heated chambers, are especially important in the aerospace sector. Autoclaves typically can reach temperatures over 240°C within 45 minutes and do so with a good accuracy (less than 0.9°C of temperature variation inside the chamber). They can also be pressurized up to 10 bar, further supplemented by the use of vacuum bags (Figure 5.6). In modern aerospace manufacture, the use of autoclaves is essential to produce defect-free composites and bonded joints.



Figure 5.6. Scheme of an autoclave used to manufacture components of bonded and composite construction.

The process time, temperature and the pressure are the most important parameters that should be controlled during the hardening process. Besides the exposure to heat, other sources of energy, such as radiation, and moisture can also be used to harden adhesives, requiring specific equipment such as ultraviolet lamp lights or radiation sources. In two-part adhesives, the hardening process does not necessarily require exposing the adhesive to heat, although higher temperature can generally be used to accelerate the process. Regarding form, paste adhesives generally have a lower curing temperature than film adhesives, which is advantageous for bonding substrates of dissimilar coefficients of expansion.

A well-known technique employed for localized hardening in the aeronautical sector is the use of heated blankets. As shown in Figure 5.7, a blanket containing resistors is heated up by passing an electrical power through it. The supplied electrical power is controlled to provide sufficient heat for local hardening of the structure or of the repair patch. These blankets are made to be quite flexible which makes them suitable for operating on curved structures such as those found in airframes.



Figure 5.7. Localized heating setup used for local heating of bonded airframe repairs.

QUALITY CONTROL OF THE ADHESIVE BONDING PROCESS

# 6. QUALITY CONTROL OF THE ADHESIVE BONDING PROCESS

We have seen that adhesive bonding is extensively used in high performance aeronautical structures such as composite components, wing structures and fuselages. However, the structures containing these bonded joints should meet established and stringent aeronautical certification standards, which always require joints to be subjected to extensive quality assessment and inspection activities. These evaluations should be carried out in a manner that ensures a durable and reliable bond, one that guarantees the integrity of the structure during service. For this purpose, destructive and non-destructive techniques might be employed immediately after manufacturing to manage the quality of the joints while in-service checks must rely solely on non-destructive inspections. Thus, bonded joints for aerospace applications are subjected to multi-step quality control programs.

In this chapter, all these assessment procedures are described in some detail, including the quality control of the incoming materials, quality assessments of the manufacturing procedures, and the destructive and non-destructive test approaches used for qualification of bonded joints employed in the aviation industry.

# 6.1. QUALITY CONTROL OF THE INCOMING MATERIALS

The first step to ensure that the bonded joints meet the appropriate airworthiness requirements occurs long before any joint is manufactured, with the qualification of the incoming materials. Quality control of the incoming materials includes an analysis of the mechanical properties of adhesive and the adherend, viscosity analysis of the adhesive, surface energy assessment and surface roughness measurement of the adherend. Dogbone shaped bulk tensile specimens and TAST joints, for example, can be used analyse the tensile and shear properties of the adhesives. For some aeronautical applications where crack propagation in service should be limited to a certain size, the fracture behaviour of the incoming materials should also be analysed by conducting standardized fracture mechanics testing procedures.

Aluminium alloys and composites are among the most commonly used adherends in lightweight aeronautical structures, such as airframes and wings. Due to manufacturing issues or handling problems, these adherends may become contaminated or even damaged and, depending on the type of the adherend (composite or metal), different tests (tensile, bending, etc) can be employed to detect these alterations. It should be noted that composite components generally require more detailed inspection procedures than those of metallic nature.

## 6.1.1. Glass transition temperature

As we have seen in Chapter 4, the  $T_g$  is an important parameter that is essential to define the service temperature of an adhesive. Measuring  $T_g$  is also an alternative approach to control the hardening quality of the adhesive materials. The use of DSC (Differential Scanning Calorimetry) and DMA (Dynamic Mechanical Analysis) are the two common methods used for this purpose. Regardless of the approach used, please note that the determined  $T_g$  value should never be assumed to correspond to a precisely defined temperature but instead as a temperature range in which the transition occurs.

In a DSC analysis, the  $\rm T_{\rm g}$  of the adhesive is related to the baseline inflection region shown in Figure 6.1.





The DMA approach is generally more sensitive than the DSC methodology. In this approach, the  $T_g$  corresponds to the peak of the tan  $\delta$  curve, the peak of the loss modulus curve E'', and the point where a sigmoidal change in the storage modulus E' is observed. All these points are shown in Figure 6.2.



Figure 6.2. Determination of the glass transition temperature of an adhesive by identifying the temperature at which a peak in damping occurs (DMA analysis).

### 6.1.2. Surface state

Wettability (or surface energy) is another critical factor in qualified bonded joints. As we have seen in Chapter 3, the lower the wettability, the higher the risk of the interfacial failure and the lower the joint strength will be, especially when adhesive joints are used for repair purposes. This reduction in adhesion can be caused by contamination due to the presence of fluids (hydraulic fluids or fuel for example) or other types of contamination. Dyne pens are very simple tools that can be used to quickly analyse the surface energy of the adherends. Using a specific marker, a line is drawn on the adherend surface and a breakage of the lines into smaller sections shows that the surface energy is lower than what has been specified by the grade of the used marker. Directly measuring the contact angle of the adhesive is another way to ensure an adequate surface energy of the adherends. The higher the contact angle, the lower the surface energy and consequently the lower the adhesion quality will be (see Figure 6.3).



Figure 6.3. A schematic representation of the equipment used for measuring the contact angle of a drop on an adherend surface.

# 6.2. QUALITY CONTROL OF THE MANUFACTURING PROCESS

After ensuring that the adhesives and substrates are within the desired specifications, most of the quality control assurance of the manufacturing process is concentrated on three substeps, comprising adhesive application, joint assembly, and adhesive hardening. Adhesives should be applied using clean tools and equipment in clean room conditions, ideally with controlled temperature and relative humidity levels, allowing for greater repeatability of the obtained results. It usually recommended to ensure that the temperature is between 18 and 32°C and that the relative humidity is kept between 20 and 65%.

We have already seen that film adhesives are extensively used in aeronautical applications, in part because the challenges associated to the application process will be lessened. However, other types of adhesives (paste adhesives for example) still have a place in airframe construction but demand greater control in its application. Controlling the amount of adhesive and the adhesive pattern on the substrate surface are the two parameters that must be controlled, ensuring that no air is entrapped in the adhesive layer. After the adhesive is applied, checks should be carried for joint alignment, adhesive thickness, applied pressure and temperature, since these have a drastic effect on the joint performance.

Lastly, the evolution and successful completion of the hardening process of an adhesive can be scrutinised using DSC (differential scanning calorimetry), FTIR (Fourier-transform infrared spectroscopy), and DMA (dynamic mechanical analysis) techniques, although this cannot be carried out directly during the curing process. Instead, samples should be taken from the assembly lines and controlled in laboratorial conditions. Still in the subject of hardening, it is also highly recommendable to precisely determine the effective temperature acting on the adhesive layer during the curing process and not rely on indirect measurements (for example, the temperature shown in an oven controller). This will ensure that the adhesive is subjected to the precisely desired curing condition.

# 6.3. QUALITY CONTROL OF FINISHED BONDED STRUCTURES

A bonded joint can be qualified for service using both destructive and non-destructive tests. In destructive tests, a flaw may initiate and or propagate within the joint. Destructive tests are usually tests of a mechanical nature where the joint will be tested until it fails. On the other hand, in a non-destructive analysis the objective is to detect the presence of specific types of defects (depending on the test technique), greater than a specific size. The following sections briefly review the different types of defects found in adhesive joints and discuss the most common destructive and non-destructive tests.

# 6.3.1. Types of defects present in bonded joints

Some of the most common defects found in bonded joints are shown in Figure 6.4. The type, size, and location of the defects should be detected with a suitable quality assessment technique. Then, according to applicable standards, a qualified technician should decide if the detected defect (or defects) can significantly change the performance of the joint or determine if the joint can sustain the necessary service loads until the next inspection occurs. Although the use of both destructive and non-destructive methods is common in airframe design and manufacturing processes, non-destructive methods are almost exclusively used during service.



Figure 6.4. Typical defects of the adhesive layer in an adhesive joint.

Almost all of the defects shown in Figure 6.4 are detectable using the existing NDT techniques but detecting weak adhesion is still a very challenging prospect. An improper or insufficient surface treatment methodology can lead to weak adhesion, a state where the adhesive and adherend are physically in close contact but there is no load bearing capability. Cracks are usually products of residual stress caused by shrinkage of the adhesive and/or the mismatch between the thermal expansion coefficient of the adherends and the adhesive (in cases where a heat curing adhesive is used). Voids are introduced during the manufacturing process, especially when the two parts of a two-component adhesive are mixed together. An incorrect adhesive application pattern can introduce voids into the adhesive layer. Porosities are formed due to the chemical reaction associated to the hardening process. While some of these defects only appear during manufacturing, some of these flaws can also appear during service conditions, which requires the implementation of additional checks. The following sections discuss the most common destructive methods used by airframe manufacturers to qualify bonded joints.

# 6.3.2. Destructive tests

### 6.3.2.1. Proof tests

For critical applications, where the failure of the joint will lead to a catastrophic failure, bonded structures should first pass a proof test where they are subjected to loads higher than those encountered during service. Although proof tests may not lead to structural failure, this still might occur if any of the joints present is not up to the desired standards and these tests are thus still classified as destructive tests. If no failure accrues in a proof test, then we can say that the bonded structure has successfully passed this test. Aircraft wings are an example of a component where a proof test (typically equal to 150% of the expected service load) is employed (as shown in Figure 6.5). It should be noted that the effects of time dependent damage such as ageing degradation or fatigue cracks are not captured in regular proof tests. Durability of the joint is often determined via specific testing procedures or by carrying out a proof test in a joint which has been subjected to ageing or fatigue loads.



Figure 6.5. Proof test of the X-47B unmanned aerial vehicle (Northrop-Grumman).

#### 6.3.2.2. Fractography analysis

After a joint has been tested to destruction, fractography techniques can be used to determine, *post-mortem*, the nature of the defect which has caused the damage and failure. Defects such as those shown in Figure 6.4 are mostly detectable with a fractography analysis of the fracture surface. Although a simple visual check is an approach commonly used to assess the status of the surface, more advanced analyses rely on instrumented methodologies, such as scanning electron microscopy (SEM) analysis, energy dispersive

X-ray spectroscopy (EDS) method, and atomic force microscopy (AFM) approach should be considered.

The failure mode (adhesive or cohesive, as shown in Figure 6.6) and failure type (brittle vs. ductile) are also detectable in a fractography analysis (Figure 6.7). A higher density of micro cracks is typical of a brittle failure, in which cracks tend to propagate perpendicularly to the direction of the maximum principal stress. In contrast, in a more ductile failure, whitening bands can be observed on the fracture surface of the joint, evidence of plastic deformation.



Figure 6.6. Example of a fractographic analysis showing the presence of a thin layer of adhesive typical of a cohesive failure close to the interface.



Figure 6.7. Scanning electron microscope images of a failure surface, showing the exposed base metal (light grey) and the adhesive (dark grey).

## 6.3.3. Non-destructive testing (NDT)

NDT procedures are essential tools to implement a fracture control plan in the aviation industry. In such plans, depending on the design method, each defect is allowed to grow to a specific size and should always be detected before reaching its critical dimension, whereupon the component is retired from service or repaired. However, for some adhesives and for some types of defects, the critical size is very small, in the order of a small fraction of a millimetre. In this case, NDT techniques still might struggle to detect such a small defect. Figure 6.8 shows a typical curve on the possibility of detection as a function of crack size. It should be noted that regardless of the defect size, the probability of detection never fully reaches one. This means that there is always the possibility of the presence of defects that are not detected by the available non-destructive inspection methods.



Figure 6.8. Probability of defect detection as a function of defect size.

The critical size of the crack, which must never be reached during service, should be defined as a function of the time between the inspections. The higher this interval, the shorter is the permissible defect size. Figure 6.9 exemplifies how the defect size growth progresses as a function of flight hours. Longer time intervals between inspections can reduce the maintenance costs but NDT techniques are limited in the size of the defects that they can detect. Furthermore, an additional factor to this analysis is the density of the defects should be analysed. The higher the density of the defects, the lower will be the allowable critical size and the higher the probability of joint failure, which demands NDT methodologies which are also able to determine the number of defects with good accuracy.



Figure 6.9. Defect growth and the inspection program required to detect the defect.

Various NDT methods are suitable to evaluate the integrity of bonded joints, ranging from the simple visual inspection to highly specialized methods such as acoustic emission or the use of Lamb waves and impedance spectroscopy. The most important of these are presented in the following sub-sections.

#### 6.3.3.1. Visual inspection

One of the earliest and easiest to implement NDT techniques, visual inspection can be performed with the naked eye or aided with simple instruments, like magnifying glasses, and requires a proper examination space with good luminosity, appropriate tools and unobstructed access to the joint. This technique is appropriate to identify defects located at the joint surface but can also be used to detect deviations in the geometrical and dimensional parameters. Since this procedure relies on human judgement, it can be relatively unreliable and subjective, which can be partially mitigated by solid training process and the adoption of strict defect classification procedures.

This method usually tends to be a first step in a more complex procedure for quality control of bonded structures, which will eventually determine if the assembly is suitable for service or if further evaluation is required. Examples of typical defects and manufacturing errors which can be identified using visual inspection are shown in Figure 6.10.



Figure 6.10. Identification of defects in bonded joints via the observation of the spew fillet.

#### 6.3.3.2. Tap test

In this simple NDT method, a hammer is used to tap the surfaces of the adherend (Figure 6.11). The sound waves resulting from the tap are influenced by the presence of a defect, such as a void or a crack. When a defect is present, the generated sound is higher pitched and more resonant, while, in a pristine joint, the tapping action creates a dull sound. This approach is surprisingly effective at detecting damage, but provides no additional information about its size, location or severity.



Figure 6.11. Schematic representation of the tap test and its operation principle.

An automated version of this test exists, where a solenoid driven impactor generates well--controlled taps in the same area, yielding more consistent results, since it captures data on the applied load and energy. This can help determine the quality of the structure under examination.

#### 6.3.3.3. Ultrasonic test

In ultrasonic testing, a special probe generates high frequency sound waves that travel through the bonded structure. When a sound wave reaches a defect, such as a void or a

crack, part of it is reflected backwards, while the remainder continues as a transmitted wave. The difference between these two waves is registered in a data acquisition system and will be interpreted by trained technician to determine if damage is present. Figure 6.12 shows this procedure schematically.



Figure 6.12. Schematic representation of the ultrasonic test and its operation principle.

Voids, porosities and cracks can be detected with this technique. However, weak adhesion is very hard to detect with ultrasonic NDT. Furthermore, determining the type and size of defect is also difficult using this approach.

#### 6.3.3.4. Radiography test

In this methodology, a source of radiation is used to emit X-ray radiation inside a closed chamber with the bonded component. This radiation, which is of high frequency and short wavelength (10 pm to 10 nm), is made to pass through the component being tested. During exposure, part of the X-rays is absorbed, while the remaining radiation is transmitted and reaches a film or sensor, located opposite to the radiation source. This film/sensor absorbs the remaining radiation, creating an image where defects in the adhesive and the undamaged material of the adherends are easily distinguishable. This occurs because the percentage of transmitted and absorbed radiation changes with variations in material properties caused by damage. With this method, one can detect and visualize cracks, voids and disbonds.

Alternatively, one can also use gamma radiation since it is also of high frequency and short wavelength (10 pm and lower). The major difference between these two variants is the source of radiation: X-rays are artificially produced with a generator, while gamma rays can only be emitted by a radioactive material source.

## 6.3.3.5. Thermal infrared method

In the thermal infrared method, also known as infrared thermography, a source of highly concentrated energy, such as a photographic flash or a halogen lamp, induces a heat pulse on the joint, causing a sudden and transient increase in temperature. The temperature distribution in the component will be influenced by the presence of a given defect since the cooling process will not be uniform. The resultant temperature distribution can be registered by means of a thermographic camera and an analysis of the images should be conducted in order to decide if the joint is qualified for service or not. Figure 6.13 details the key elements of this procedure.

The infrared thermography method is classified as a non-contact NDT approach, making it especially useful to test joints of hard accessibility. Cracks, void, insufficient hardening and adhesion issues are some of the defects detectable by this technique.



Figure 6.13. Schematic representation of the thermal infrared method and its operation principle.

#### 6.3.3.6. Acoustic emission test

In this method, sensors placed on a free surface of an adherend are used for detecting the sound or vibration caused by small excitations, i.e., small impacts, crack propagation, etc. These signals are compared with reference signals generated by a pristine structure, allowing to detect damage, since changes in the propagation of sound waves can be caused by the emergence or growth of a defect. Additionally, this technique is able to detect delamination or fibre breakage in composite adherends and some studies have even found that weak adhesion in the interface layer can also be detected. This procedure is shown schematically in Figure 6.14.



Figure 6.14. Schematic representation of the acoustic emission method and its operation principle.

Some authors consider this technique as a destructive test, given that the specimen needs to be mechanically loaded to generate the necessary waves and vibrations, although the monitored structure is still expected to be fit for service after the test is completed. In fact, just the act of generating an excitation signal with a small load or impact has the potential to induce damage (however minor) in the structure.

#### 6.3.3.7. Lamb wave method

Lamb waves are a type of ultrasonic elastic wave that propagates through a bounded medium such as a bonded structure, generating multi-modal and dispersive waves. Given their complexity, these waves can be subdivided into symmetric quasi-axial waves, or S- waves, and antisymmetric quasi-flexural waves, or A-waves. These exist simultaneously, making their propagation extremely complex in nature and difficult to process. In Lamb wave-based NDTs, a signal generator generates a transient electric signal that is sent to a piezoelectric actuator, which in turn converts the signal into ultrasonic Lamb waves. When the generated waves reach a given defect, these are partially reflected and partially transmitted, similarly to what occurs in traditional ultrasonic waves. Piezoelectric sensors convert the received waves into an electric signal which is then read by a data acquisition system and a computer for signal processing (Figure 6.15).



Figure 6.15. Schematic representation of the lamb wave method and its operation principle.

This method is currently the target of active research and has already shown promising results in detecting various types of damage such as voids or weak adhesion. However, it requires complex signal processing tools and algorithms, which still limits its widespread applicability.

### 6.3.3.8. Electromechanical impedance spectroscopy

Electromechanical impedance spectroscopy (EMIS), used as an NDT approach, uses both the mechanical and electrical concepts to detect defects in bonded structures. Similar to the lamb waves method, EMIS also employs piezoelectric sensors which can act both as an actuator and as a signal receiver (sensor). However, in contrast with the lamb wave technique, where a set of waves is generated only for a given excitation frequency, EMIS excites the joint in a much wider range of frequencies.

In the EMIS approach an AC voltage is first generated using a generator and an electrical signal is delivered to a piezoelectric component which acts as an actuator. Piezoelectric materials can convert electrical signals into a mechanical excitation, and vice versa since these sensors have a coupled electromechanical behaviour. This means that they can also receive the mechanical response and convert it to electrical signals. The obtained electrical signals are sent to a data acquisition system where the response signals are recorded and processed. The general scheme of the EMIS set up is shown in Figure 6.16. The piezoelectric sensor can be permanently bonded to the structure for a continuous health monitoring of the bonded structure in service. Compared to other NDT techniques, EMIS is a lightweight and an inexpensive approach with a wider range of frequencies than can be used in this method.



Figure 6.16. Schematic representation of the electromechanical impedance spectroscopy method and its operation principle.

Using the EMIS technique, it becomes possible to detect adhesive disbonding, and cracks in adhesive joints. As we have seen, weak adhesion between the adhesive and the adherend is quite difficult to detect but research on the EMIS technique suggests that this technique will be able to so.

#### 6.3.3.9. Laser-based NDT

In laser-based NDT, laser signals are transmitted through fibre-optic cables to special fibre heads. These generate ultrasounds through non-contact means and travel freely through the adhesive joint. Another fibre head receives the incoming ultrasound wave and generates another light signal that is sent through a fibre-optic cable for signal processing (as shown in Figure 6.17). This NDT method is almost exclusively used in the aeronautical industry. Boeing has pioneered the use of this method to detect weak adhesion in manufactured airplane components. However, the generated loads may be strong enough to damage defect-free areas and cause immediate joint failure in bondlines where some defects are already present.



Figure 6.17. Schematic representation of a laser-based NDT method and its operation principle.



# 7. SAFETY AND REPAIR

The use of adhesives in the aerospace sector has increased significantly in recent decades, but this growth has also exposed the workers tasked to build modern aircraft to new hazardous materials and dangerous working conditions. Furthermore, these processes can also generate a large volume of industrial waste, which must now be disposed of properly, without risk to the environment. In addition, reparability and recycling are also particularly sensitive issues in adhesive bonding, since adhesively bonded components are not particularly easy to maintain, service and recycle. Faced with international guidelines forcing ever greater recyclability of materials used in the transportation sector, these characteristics may represent an obstacle to a wider application of adhesives in aeronautical applications. However, new procedures and materials are constantly being developed to further the reuse and repair of bonded components. This chapter offers an overlook into the main safety risks associated to adhesive usage and also provides an insight into the repair procedures that can be used to extend the life of components used in the aeronautical sector.

# 7.1. GENERAL PRECAUTIONS ASSOCIATED TO ADHESIVE USAGE

Before we delve in the specific hazards of different types of adhesives, the general precautions for the safe handling of adhesives should be discussed. These include the use of appropriate protective equipment and correct interpretation of the warnings and documentation supplied with these materials. Work should never commence without the operator reading and understanding the relevant technical and safety data sheets (Figure 7.1). The operator should know how to interpret and evaluate the health and safety hazards for the different stages of the joint production process, select the appropriate protective equipment and design a production process that minimises exposure to risk.

Product packaging also features information labels with very summarized and specific information, describing the level of danger of the products. This information is condensed into easy-to-read pictograms, highlighting the main risks associated to the product and indicating the actions to be taken in case of direct contact with the product.



Figure 7.1. Examples of safety datasheets of adhesives used in aerospace applications.

### 7.1.1. Use of personal protective equipment

The use of personal protective equipment (PPE) is essential to protect those who operate with adhesives from the hazards associated with adhesives and all other equipment and products involved in the bonding process. Many different pictograms alert the user on the required PPEs for a given task, such as those shown in Figure 7.2.



Figure 7.2. Pictograms of the most important types of personal protective equipment.

Many tasks associated with adhesive bonding work in the aerospace sector have the potential to cause serious eye injuries. Thus, the use of safety glasses is mandatory in operations where a risk of this nature has been identified. The market offers a wide range of safety glasses, and the choice of the suitable configuration will depend primarily on the nature of the risk and of the tasks to be carried out.

In the aerospace sector, manufacturing rates are generally low and much of the adhesive application work is still manual in nature, exposing the operators' hands to various hazards and making the use of appropriate safety gloves essential. The most commonly used gloves for handling chemicals, such as adhesive, primers and etching solutions, are nitrile and natural latex gloves, as these ensure a high level of flexibility and resistance against most chemicals. When surface preparation is carried out with shot blasting, PVC gloves are recommended, as they provide excellent protection against abrasion. Heat resistant gloves are mandatory when the work is carried out at extreme temperatures.

Safety shoes are another essential personal protective equipment for bonding operations in an industrial environment. These consist of a durable leather upper, an insole to absorb the impacts caused by the wearer's body weight, and the sole, which must offer abrasion resistance, electrical insulation and protect from contact with toxic, chemical and hot substances.

Some substances used in adhesive bonding processes release harmful and volatile substances that will become airborne and easily inhaled by workers, even if masks and other breathing filters are used. Therefore, the air quality in industrial environment dedicated to bonding must always be controlled to ensure the well-being of workers. To ensure air quality and ensure that the level of volatile compounds (usually abbreviated as VOC) does not reach toxic values, the workplace must be adequately ventilated and/or equipped with devices that ensure air replenishment and filtration. Figure 7.3 shows a filtration system suitable for operation with adhesives and other products associated with this process.



Figure 7.3. Laboratory equipped with extraction systems suitable for handling the volatile emissions of adhesives and other chemical used in the bonding process.

# 7.2. DANGERS ASSOCIATED TO ADHESIVES USED IN AEROSPACE APPLICATIONS

# 7.2.1. Epoxy adhesives

As mentioned repeatedly in this book, epoxies are among the most widely used adhesives in structural applications in the aerospace sector. Considering a two-component epoxy adhesive (hardener and resin), the resin is generally the component that presents the most serious associated risks. Epoxy resins in liquid or paste form (uncured) are generally irritating to the eyes and skin and toxic to aquatic organisms. The main hazards associated with epoxy adhesives are the exothermic hardening reaction and the phenomenon of sensitisation to the resin. The first of these is minimised by mixing the adhesive components in the quantities and proportions recommended by the manufacturer in the data sheet and by avoiding direct contact with the mixture. On the other hand, the sensitisation process is an allergic reaction that develops after repeated skin contact with the epoxy resins. This reaction may occur several days after contact with the material and usually takes the form of dermatitis in the areas where contact has occurred.

## 7.2.2. Phenolic adhesives

Phenolic adhesives contain diverse chemical compounds such as phenol and formaldehyde. Inhalation and exposure to phenol is extremely irritating to the skin, eyes and mucous membranes. Excessive exposure may lead to irregular breathing, weakness, tremors, loss of coordination, convulsions and even respiratory arrest. Formaldehyde, a key component of these adhesives, is also a harmful substance. When present in the air at levels above 0.1 ppm, some individuals may suffer from irritation in the eyes, nose and throat. In this regard, the technical data sheet for this adhesive should be read carefully and the necessary measures should be implemented, given the high level of risk presented.

## 7.2.3. Aromatic adhesives

A wide range of aromatic adhesive formulations are available, the most common of which are the polyamides, bismaleimides, and polybenzimidazoles. Although the dangers associated to each of these formulations can vary, many of the issues associated to aromatic adhesives stem from the solvents present to facilitate their usage. These highly volatile solvents can cause dizziness, nausea, fatigue, lack of coordination and even coma. The probability of accidents increases under the influence of these compounds and long-term exposure may eventually lead to brain damage. The application of this adhesive should take place in a well-ventilated environment, supported with adequate PPE.

### 7.2.4. Silicones and sealants

Synthetic rubbers are relatively stable and safe compounds. However, in their uncured state they are known to cause skin and eye irritation on contact and thus should always be handled with gloves. Furthermore, some types of room temperature vulcanizing silicones emit acetic acid during the curing process, which is corrosive in high concentrations.

## 7.2.5. Other materials associated to bonding processes

One of the most dangerous phases in surface preparation processes is the degreasing of the surfaces to be bonded, which usually require the use of highly toxic and corrosive products, such as acid solutions (sulphuric, nitric, etc.), volatile solvents (trichloroethane, chloroform, carbon tetrachloride, trichloroethylene, etc.) and alkaline solutions (cyanides, borax, sodium silicates, etc.). The constituents of the surfaces to be prepared also require special attention in their handling. For example, plasticisers can be released from some polymeric materials and polymer matrix composites, with ecological and functional implications. In the surface preparation and coating processes of metals for airframe construction, such as duralumin, it is also common to encounter electrostatic baths that can use harmful solutions. Moulds and jigs are also usually coated with powerful release agents, which are known to release very toxic solvents and reaction products. These release agents should always be applied in highly ventilated locations, in order to avoid exposure to these chemical compounds.

In more energetic processes, especially those where specialised equipment is used, the potential risks also increase and are usually associated with thermal exposure, vibration and high electrical voltages. This can lead to cutting, bruising and crushing hazards.

# 7.3. REPAIR

One of the most important uses of adhesives in the aeronautical industry is in the bonding of repair patches to damaged airframes or components, applied in an effort to extend the service life of these structures. Figure 7.4 schematically shows a repair patch bonded on the cracked section of a fuselage panel.



Figure 7.4. Schematic representation of an adhesively bonded repair patch.

## 7.3.1. Performance of bonded repairs

Bonded repairs can recover the strength of a damaged structure or even increase its durability. However, despite the outstanding performance shown by repair patches bonded to metallic and composite airframes but still there are some concerns on the damage tolerance of bonded repairs. Figure 7.5 shows the extension in service life that might be achievable by repairing a cracked airframe skin. A bonded patch can serve as an effective crack stopper and can considerably reduce the crack growth rate in the parent structure.



Figure 7.5. Extension of the service life gained with the use of bonded crack stoppers.

While a bonded component manufactured in a highly controlled industrial process generally exhibits very high reliability, the performance of bonded repairs is less consistent, since they are highly dependent on the skill of the bonder and on the conditions available to carry out the bonding process. Accordingly, despite the significant extension of the service life of the damaged parts that can be achieved by using bonded repairs, regulations applicable to repairs tend to follow a conservative perspective, where the eventual improvements brought by the bonded repair are never considered as a positive contribution to the life span of the damaged part.

#### 7.3.2. Repair procedures

Carrying out repairs is a challenging activity and fraught with danger for an inexperienced bonder who does not follow standardized repair procedures. Issues such as an insufficient surface preparation, excessive degradation of adhesives due to the environmental ageing and contamination and the presence of porosity in the bondline are the most common reasons for the failure of bonded repairs in aeronautical applications.

Although the composition of primary and secondary structures is evolving quickly, with increasingly larger amounts of composite materials, repair procedures generally follow the same methodology, irrespectively of the base materials and the role of the component being worked upon.

The following steps are undertaken in a repair process:

- An inspection is carried out, assessing the nature and extent of damage;
- The damaged material is removed as far as possible;
- Contaminated material is removed or cleaned;
- The repair area is prepared, be it with surface treatments or mechanical modifications;
- New load bearing material (a repair patch) is installed in the damaged area;
- A final inspection is carried out to identify unseen defects;
- If necessary, a surface finish restoration is carried out.

In the bonded repair of metal structures (mostly aluminium alloys), repair patches are usually composed of composite materials, since these offer very large specific strength and stiffness, minimizing the amount of material used in the patch and thus the weight added to the airframe. However, the differences in the thermal expansion coefficients between the patch, the adhesive, and the metal of the airframe can induce significant thermal stresses in the adhesive layer, all of which have the potential to significantly reduce the durability of the adhesively bonded repair. Furthermore, since airframes experience a wide range of temperatures in service, with large variations during each flight, the damaging effect of the residual stresses can increase and accumulate due to these cyclic environmental conditions.

In case of all-composite fuselages or components, the issue of thermally induced stresses in repair patches is much lower. However, in this case the repairs are much more complex in nature, since the damage mechanism are usually complex (for example, the laminate splitting due to impact loads and hole damage shown in Figure 7.6) and the repair techniques must consider the layered construction of the composite adherends and repair this layup accordingly.



Figure 7.6. Examples of types of damage generated in composite structures.

In the case of simple laminate panels, repairs are usually performed with the assistance of a film adhesive. The damaged area is first bored out and cleaned and the filler plies of a prepreg composite are inserted in the void, supported by a backup ply. Alternatively, a tapered hole is created, which is filled with successive layers of repair plies, topped by a final, extra ply. Vacuum bags and heated blankets can then be applied over these repairs in order to harden both the film adhesive and the prepreg composite plies These methodologies are shown schematically in Figure 7.7.



Figure 7.7. Repair process of a laminate panel.

Should the structure to be repaired consist of a composite honeycomb panel, the repair procedure is slightly different. These processes usually start with the insertion of a new section of replacement core in the honeycomb, which is bonded into place using a core

splice adhesive, which is generally a low strength adhesive since it will face very limited loads during service. After this process is carried out, the process of repairing the skins of the honeycomb follows a procedure generally similar to that described above for laminate panels. In this case, while one of the skins is repaired with a film adhesive and repair plies, the other side, bearing a hole, must be supported by some sort of temporary mould and a packing piece, avoid the release of the newly inserted core as a result of the pressure applied by a vacuum bag. This process is shown in Figure 7.8.



Figure 7.8. Examples of repair processes for bonded honeycomb panels.

An important topic related to repair activities is the detection of defects. We have previously discussed the subject of weak adhesion, and this is also one of the main challenges associated to bonded repairs. Weak adhesion can significantly reduce the service life of the repair but current NDT techniques struggle to precisely detect this defect. Due to the limitations shown by the current NDT methods in quality assessments of the bonded repairs (described in Chapter 6), some regulations prescribe that the load bearing capacity of the repaired structure should be calculated based on that of the cracked component without the bonded repair been considered. However, this is usually reserved for more critical parts of the airframe, especially for primary structures where no redundancy or back up is available.




# 8. JOINT DESIGN

Joint design is one of the most complex activities associated with creating an adhesive joint and of crucial importance to aerostructures, where high specific strength is indispensable. In essence, this activity consists of selecting the materials and the geometrical parameters that ensure that the joint can withstand a specific load or set of loading conditions without failure.

## 8.1. MAIN GEOMETRICAL CONFIGURATIONS OF BONDED JOINTS

The number of geometries that can be used to create adhesive joints is very vast, with many distinct solutions. Examples of the most common joint geometries found in the aerospace sector can be found in Figure 8.1.



Figure 8.1. Typical geometrical configurations of bonded joints used in aerospace construction.

Of these, the single lap joint (SLJ) is perhaps one the most important, mainly because of its simplicity and effectiveness. We have seen in the introductory chapters that adhesives generally perform best under shear loads and the SLJ fully exploits this fact, offering an adhesive layer mostly aligned with the load direction. However, the adherends in a SLJ are not perfectly aligned, which creates a load imbalance in the adhesive layer allowing for the appearance of peel stresses at the ends of the overlap length, induced by a large bending moment.

In contrast, alternative, yet more complex geometries, such as the double overlap joint, align the load more effectively with the adhesive layers and allow it to operate in a more perfect shear state, although they are more difficult to manufacture and more expensive. The T-joint is also extensively used in aeronautical construction, being highly suited for bonding reinforcement stringers to stiffen thin and flexible fuselage panels.

#### 8.2. Analytical methods

The prediction of the performance of a bonded joint can be carried out based on analytical methods, which use mathematical expressions to calculate the stress level in adhesive joints or numerical methods, which use more complex models to calculate stress and displacement fields as a function of the boundary conditions applied to geometrical models of the adhesive joints. Analytical models are very easy to implement in calculation processes and software. For example, the JointDesigner tool (www.jointdesigner.pt) offers different analytical methods to calculate joint strength in adhesive joints.

## 8.2.1. Simple analysis

Analytical methods were the first used to determine the strength of adhesive joints, still in the first half of the 20th century, borne out of research work carried out in the aeronautical field. These models generally consist of mathematical expressions that relate the joint geometry and its loading to the material properties and allow the determination of the stresses acting on the adhesive layer. These stresses can be compared to a given limit (for example material strength) and if exceeded, the joint is expected to fail. Some analytical models provide multiple calculation pathways and can determine if the failure will occur in the adhesive layer (cohesive failure of the adhesive) or in the adherend (cohesive failure of the adherend). To simplify this presentation, we will first compare the stress calculation capabilities of the different analytical models for SLJs and only later discuss the appropriate failure criteria for different materials.

The model shown in Figure 8.2 assumes that the entire adhesive layer is being uniformly loaded (so the resistant area is effectively the entire bonded area). It also assumes that the shear stress acting on the adhesive layer can be calculated directly from the load acting on the adherends (marked as P in Figure 8.2).



Figure 8.2. Determination of the average shear stress acting on a single lap joint.

This model assumes that the adhesive does not deform (neither elastically nor plastically) and that there is a uniform and constant distribution of shear stress along the adhesive layer, which is simply not true for all adhesives. In fact, this only occurs for adhesives which are very flexible and ductile, which limits the applicability of this model for most structural applications

#### 8.2.2. Volkersen

In 1938, Volkersen, carrying out research in bonded joints for aeronautical structural applications, found that the shear stress acting on the adhesive layer is not evenly distributed. In simple terms, Volkersen was the first to discover that in a loaded joint with deformable adherends, the adhesive layer will also have to deform to keep up with the adherends but since the ends of the adherends are more heavily loaded, a strain differential is created, which forces the adhesive to deform unevenly as well. As a result, it is at the ends of the overlap length that the shear stress reaches its maximum value and the adhesive will first fail. Figure 8.3 shows this effect schematically, demonstrating that the resulting shear stress distribution will have peaks at the edges of the overlap.



Figure 8.3. Differential straining of a bonded joint and the respective shear stress distribution, as reported by Volkersen.

The Volkersen model allowed to conclude that an excessively rigid adhesive should never be used in structural applications, since it cannot support the natural deformation of the adherends without highly becoming stressed.

The implementation of the Volkersen model follows the geometrical parameters listed in Figure 8.4. Its calculation uses the overlap length (/), the thickness of the adherends ( $t_1$  and  $t_2$ ), the adhesive layer thickness ( $t_a$ ). As for mechanical properties, since Volkersen's model is purely elastic, it only requires the modulus of elasticity of the adherends ( $E_1$  and  $E_2$ ) and the shear modulus of the adhesive ( $G_2$ ).



Figure 8.4. Geometrical parameters of the joint and the free body diagrams used in the Volkersen model.

According to Volkersen, the distribution of shear stress in the adhesive ( $\tau$ ) as a function of the applied load (*P*) can be obtained by carrying out a balance of the loads acting on the upper adherend:

$$\sigma_1 bt_1 + \tau bdx = (\sigma_1 + d\sigma_1) bt_1 \Rightarrow \frac{d\sigma_1}{dx} = \frac{\tau}{t_1}$$
**8.1.**

This will be followed by the balance of forces acting on the bottom adherend:

$$\sigma_2 bt_2 = (\sigma_2 + d\sigma_2) bt_2 + \tau bdx \Rightarrow \frac{d\sigma_2}{dx} = -\frac{\tau}{t_2}$$
 8.2.

The balance of the joint is achieved when:

Shear deformation in the adhesive:

$$\gamma = \frac{\tau}{G_a}$$

$$\gamma = \frac{1}{t_a} (u_1 - u_2)$$

$$\Rightarrow \frac{d\gamma}{dx} = \frac{1}{G_a} \frac{d\tau}{dx} = \frac{1}{t_a} \left( \frac{du_1}{dx} - \frac{du_2}{dx} \right) = \frac{1}{t_a} \left( \frac{\sigma_1}{E_1} - \frac{\sigma_2}{E_2} \right)$$

$$8.4.$$

From Equations 9.1, 9.2 e 9.4:

$$\frac{1}{G_a} \frac{d\tau}{dx} = \frac{t_1}{G_a} \frac{d^2 \sigma_1}{dx^2} = -\frac{t_2}{G_a} \frac{d^2 \sigma_2}{dx^2} \Longrightarrow t_1 \frac{d^2 \sigma_1}{dx^2} = \frac{G_a}{t_a} \left( \frac{\sigma_1}{E_1} - \frac{\sigma_2}{E_2} \right)$$
**8.5.**

From Equation 8.3:

$$\sigma_2 = \frac{P}{bt_2} - \sigma_1 \frac{t_1}{t_2}$$
 **8.6.**

Replacing Equation 9.6 in Equation 9.5:

$$\frac{d^2\sigma_1}{dx^2} - \lambda^2\sigma_1 + C_0 = 0$$
**8.7.**

where

$$\lambda^{2} = \frac{G_{a}}{t_{a}} \left( \frac{1}{E_{1}t_{1}} + \frac{1}{E_{2}t_{2}} \right)$$
$$C_{0} = \frac{G_{a}}{t_{a}} \frac{P}{bE_{2}t_{2}t_{1}}$$

The solution for Equation 9.7 is of the type:

$$\sigma_1 = A \cosh(\lambda x) + B \sinh(\lambda x) + \frac{C_0}{\lambda^2}$$
 **8.8.**

With the following boundary conditions:

$$\sigma_1 = 0, \ \sigma_2 = \frac{P}{bt_2} \ \text{para } x = 0$$
 **8.9.**

$$\sigma_2 = 0, \ \sigma_1 = \frac{P}{bt_1} \ \text{para } x = l$$
 **8.10.**

Using Equations 9.9, 9.10 e 9.8, we can determine the *A* and *B* constants:

$$A = -\frac{C_0}{\lambda^2}$$
$$B = \frac{C_0}{\lambda^2} \frac{\cosh(\lambda l) - 1}{\sin(\lambda l)} + \frac{P}{bt_1} \frac{1}{\sinh(\lambda l)}$$

The solution for Equation 9.7 is:

$$\sigma_{1} = -\frac{C_{0}}{\lambda^{2}} \cosh(\lambda x) + \left\{ \frac{C_{0}}{\lambda^{2}} \left[ \cosh(\lambda l) - 1 \right] + \frac{P}{bt_{1}} \right\} \frac{\sinh(\lambda x)}{\sinh(\lambda l)} + \frac{C_{0}}{\lambda^{2}}$$
**8.11.**

From Equation 9.1:

$$\tau = t_1 \frac{d\sigma_1}{dx} = -\frac{C_0 t_1}{\lambda^2} \lambda \sinh(\lambda x) + \left\{ \frac{C_0 t_1}{\lambda^2} \left[ \cosh(\lambda l) - 1 \right] + \frac{P}{b} \right\} \frac{\lambda \cosh(\lambda x)}{\sinh(\lambda l)} \quad \textbf{8.12.}$$

Replacing  $C_0$  and  $\lambda^2$  and using  $\overline{\tau} = \frac{P}{bl}$ :

$$\frac{\overline{\tau}}{\overline{\tau}} = \frac{\lambda l}{\phi \sinh(\lambda l)} \left[ (\phi - 1) \cosh(\lambda (l - x)) + \cosh(\lambda x) \right]$$
**8.13.**

where

$$\phi = \frac{E_1 t_1}{E_2 t_2} + 1$$
 **8.14.**

In which:

$$\frac{\tau(0)}{\overline{\tau}} = \frac{\lambda l}{-\phi \sinh(\lambda l)} [(\phi - 1) \cosh(\lambda l) + 1]$$

$$\frac{\tau(l)}{\overline{\tau}} = \frac{\lambda l}{\phi \sinh(\lambda l)} \left[ (\phi - 1) + \cosh(\lambda l) \right]$$

Considering that:

$$\frac{E_1 t_1}{E_2 t_2} = \phi - 1$$
 **8.15.**

Where, for balanced joints,  $\phi = 2$ , i.e.  $\phi - 1 = 1$ 

We arrive at the final expression of the Volkersen model:

$$\frac{\tau(0)}{\overline{\tau}} = \frac{\tau(l)}{\overline{\tau}} = \frac{\lambda l}{2\sinh(\lambda l)} [1 + \cosh(\lambda l)]$$
**8.16.**

### 8.2.3. Goland and Reissner

Goland and Reissner created a model based on Volkersen's formulation but with the added effect of bending. The geometry of a SLJ joint is, as we have seen before, naturally unbalanced, inducing peel stresses in the adhesive layer. Goland and Reissner's formulation shows that the thicker the adhesive layer and the more severe the misalignment of the joint, the greater are the bending moments acting on the joint. Figure 8.5 shows this effect schematically.





These peel stresses arise again at the ends of the overlap, at the same location where Volkersen predicted peaks in the shear stress distribution (Figure 8.5). As such, it becomes evident that these areas are by far the most heavily loaded in an adhesive joint and are those that define the ultimate strength of a single lap joint.

A simple solution to avoid these stresses consists in the use of the double lap joint (DLJ), where the bending moments acting on the two adhesive layers are greatly reduced, since the adherends are in a more balanced state.

#### 8.2.4. Hart-Smith

The Volkersen and the Goland and Reissner models only consider an elastic behaviour for the adhesive and the adherends, which means that the stresses generated in the joint are always directly proportional to the deformation it undergoes. However, most adhesives have the capacity to deform plastically before breaking and, as a result, stresses are distributed differently along the adhesive layer during the yielding process.

Thus, Hart-Smith proposed a model that demonstrates that plastic yielding of the material at the ends of the overlap length (case B in Figure 8.6) does not necessarily mean immediate failure of the joint, since a large part of the adhesive layer can still bear additional load. Although accurate for highly ductile adhesives, the practical use of this model can be limited by the fact that it requires accurate elasto-plastic properties of the adhesive, which are often not readily available.



Figure 8.6. Additional strength provided by the plastic deformation of the adhesive, as predicted by the Hart-Smith model.

## 8.3. FAILURE CRITERIA FOR BONDED JOINTS

In the initial section of this chapter, we have presented models that provide stress or strain distributions along the adhesive layer. However, by themselves, these models are not sufficient for joint design purposes since they must be used in conjunction with an adequate failure criterion. These models are applicable to a specific set of conditions and failure criteria. As a rule of thumb, one can state that:

- Brittle adhesives: maximum stress criteria (Volkersen or Goland and Reissner)
- Ductile adhesives: maximum strain criteria (Hart-Smith)
- Very ductile adhesives (> 20% in shear): generalized yielding

## 8.3.1. Cohesive failure in the adhesive

#### 8.3.1.1. Generalized yielding of the adhesive

In brittle adhesives, which allow for a very limited level of plastic deformation, failure often occurs because the maximum shear stress of the adhesive is reached. In these cases, a purely elastic analysis, such as the one proposed by Volkersen, is perfectly adequate. If we are facing a loading where large peel stresses are generated, an approach based on the Goland and Reissner model will be more appropriate, since this model allows to simultaneously determine the distribution of the peel and shear stresses, which can be used to identify whether the adhesive reaches its maximum strength (in mode I, mode II or a combination of the two). However, as we have seen previously, in most cases adhesives undergo plastic yielding during loading, which results in failure loads that in practice are higher than those predicted by methods that only consider elastic behaviour. This limit condition is shown as case C in Figure 8.6. Figure 8.7 shows a load-displacement curve, similar to those that can be obtained by experimentally testing a specimen, which illustrates how the plastic yielding process has an important impact on the prediction of the failure load and the accuracy of the results obtained.



Figure 8.7. Demonstration of the additional failure load attained by failure criteria that account for plasticity.

A special case of the Hart-Smith model corresponds to the generalised yielding of the adhesive. In this case, the considered adhesive can be deformed uniformly, distributing the shear stresses evenly throughout the adhesive layer. This method is suitable for very ductile adhesives, i.e. those with more than 20% deformation at break when subjected to shear. The same model is also suitable for elastic adhesives (highly deformable adhesives such as one-part polyurethanes or silicones). However, in the latter case, the stress redistribution is not due to the ductility of the adhesive, but to the fact that the adhesive is highly flexible.

#### 8.3.2. Failure in the adherend

#### 8.3.2.1. Adherend yielding

In joints with metallic adherends, joint failure is often the result of plastic deformation of the metal. If the stress level on an adherend exceeds the yield strength of the metal, the joint will almost certainly fail. This does not necessarily mean that the metal adherends will rupture, leaving an intact adhesive layer in place. Instead, failure is driven by yielding of the metallic adherend, mainly near the ends of the overlap length. (as shown in Figure 8.8). The aggravated stress concentrations and the additional localised deformation stemming from adherend yielding will further damage the adhesive, eventually causing its complete failure. This type of failure is quite evident in the shape of the load-displacement curve, also shown in Figure 8.8.





Figure 8.8. Failure process of a bonded joint via yielding of the adherends.

A simple method to determine the strength of adhesive joints was proposed by Adams in 2002, simultaneously considering two conditions: adhesive failure by generalised yielding and adherend failure by plastic deformation, as shown in Figure 8.9. The adhesive yielding calculation still assumes that the shear stress is uniformly distributed along the bonded area

and thus it is more suitable for ductile and elastic adhesives. Adherend failure is analysed in a slightly more complex way, divided into two different regions. In the first, the adherend failure load is a factor of the overlap length and the adherend thickness, a correlation based on the observation of experimental data. Once the overlap length is 20 times larger than the adherend thickness, the load that will yield the adherends is assumed to be constant.



Figure 8.9. The Adams model for the determination of failure in a bonded joint, accounting both for adherend yielding and adhesive failure.

The perpendicular line in Figure 8.9 estimates the failure load of the adhesive layer in the case of a uniform shear stress. The predicted failure is obtained by multiplying the area of the adhesive layer, overlap length (*b*) times the joint width (*l*), with the shear strength of the adhesive ( $\tau_{v}$ ).

The behaviour of the adherend is, as stated above, more complex. In this case, two distinct regions are defined, arising from the theory of Goland and Reissner (Equation 9.17).

$$P = \frac{\sigma_y bt}{(1+3k)}$$
**8.17.**

The failure load is dependent on the strength of the adherend, the thickness of the adherend (*t*), the width of the adherend (*b*) and also of a *k* factor. This factor accounts for joint rotation in the relationship between the bending moment and the applied load. If the loads applied and the overlap length are small, this effect is practically negligible, and the *k* is considered to be 1. Thus, Equation 9.18 states that:

$$P = \frac{\sigma_y bt}{4}$$
 8.18.

However, if the overlap length is more than twenty times larger than the thickness of the adhesive, k can be assumed to zero. Thus, the load necessary to achieve yielding in the adherend is given by Equation 9.19.

$$P = \sigma_{v} bt$$
 8.19.

#### 8.3.2.2. Interlaminar failure of composite adherends

In cases where adhesives are used to bond composite adherends, suitable failure criteria should include an analysis of the peel stress generated at the ends of the overlap length. Peel stresses have an adverse effect on composite materials, since in practice the plies are joined together only by a thin layer of resin, as illustrated in Figure 8.10.



Figure 8.10. Interlaminar failure process of composite adherends of a bonded joint.

When composites are loaded parallelly to the reinforcement layer they exhibit extraordinary strength. However, for transverse loads (perpendicularly to the fibres), the strong and stiff fibres of the reinforcement cannot play a role. The resin matrix holding the layers together will be the weakest link in the composite layup and failure will be of an interlaminar nature process. Thus, if an adhesive joint generates peel stresses that exceed the interlaminar strength of the composite adherend, the joint will fail prematurely.

## 8.4. FAILURE LOAD PREDICTION USING NUMERICAL METHODS

The use of finite element method (FEM) is a powerful design technique that was created in the 1940s to solve complex elasticity and structural analysis problems in diverse applications, initially focused on the civil engineering and aeronautical applications. Since then, its use and

features have greatly expanded, including more advanced capabilities to model adhesives and adhesive joints.

The FEM first divides a structure into a mesh of small elements, connected by multiple nodes. Boundary conditions are applied to this mesh, mimicking the loads acting on the real component. A system of equations is then established combining the influence of each node and element and their boundary conditions. The goal of the FEM is to determine the displacement of the nodes (D), having prior knowledge of the stiffness of the body (K) and the loads and reactions acting on it (R). Thus, based on the mechanics of elasticity, an equation can be established which states that the K.D=R. Generally speaking, FEM is known for its capability to adapt to any kind of geometry and its flexibility in the calculation of stress values and fields. However, due to the time necessary to create, modify and calculate a model, it is less practical for quick parametric studies than the previously discussed analytical models. An example of a simple model of a bonded T-joint created with FEM is shown in Figure 8.11.



Figure 8.11. Example of a finite element numerical model of a bonded T-joint, showing the mesh and the deformed, stressed shape.

In a classical finite element analysis (FEA), the obtained solution is highly dependent on the mesh size. For example, a coarse mesh with large elements will average the stresses acting on a joint and erroneously show a low stress region near the edges of the overlap length of a SLJ. In contrast, a highly refined mesh (with very small elements near this critical area) will provide a more accurate calculation of the local stress field, (Figure 8.12). However, excessively fine meshes are computationally heavy and it is necessary to find a balance between a mesh that can accurately represent the true stresses acting on the joint and one that is simple enough to be computed quickly.



Figure 8.12. Influence of mesh size on the stress distribution distributions calculated with FEA (stresses taken in the middle of the adhesive layer.

However, a different issue can also arise if a mesh is refined with extremely small elements in locations of large stress concentration, as the model will output infinite stresses. Thus, if a failure criterion based solely on stress level is used, this approach will hinder the accurate prediction of joint performance and the result will be highly dependent on mesh size, as schematically shown in Figure 8.13.



Figure 8.13. Location of stress singularities in SLJs (top) and the influence of the mesh size in determining the stresses acting on these singularities (bottom).

Some approaches avoid this issue by taking stress measurements at a given distance and using these values for design purposes. However, the use of damage mechanics is a more powerful technique for solving this issue. Damage mechanics models use special elements known as cohesive elements. Cohesive zone models (CZMs) reproduce the behaviour of an adhesive using constitutive material laws which combine the elastic behaviour with a damage component. A CZM element will become damaged (lose stiffness and strength)

immediately after a certain stress level is reached, but this does not mean that the element will immediately fail. Damage will accumulate and progress until the fracture energy of the adhesive is reached. Figure 8.14 shows the basic principles of this model, showing how cohesive elements are integrated in a mesh and the cohesive zone laws that represent the material behaviour.



Figure 8.14. Principle of operation of a cohesive zone model and different cohesive law shapes.

## 8.5. PARAMETERS AFFECTING JOINT PERFORMANCE

Geometric parameters and adhesive properties all have an important effect on the mechanical performance of bonded joints. In this section the effects of the adhesive layer thickness, overlap length and adherend and adhesive properties on the performance of a bonded joint are described. Furthermore, the effect of temperature is also described.

## 8.5.1. Effect of the adhesive layer thickness

Research on the effect of adhesive layer thickness on joint performance has consistently determined an optimal value of around 0.1 - 0.2 mm for relatively rigid adhesives, such as the epoxies and aromatic adhesives used in airframe construction. Generally, the aeronautical sector operates with very tight tolerances and thus structural adhesive layers are designed and manufactured to be well within these ranges of optimum values.

Surprisingly, most of the analytical models imply than larger adhesive thicknesses lead to an increase in joint strength, since this smoothen the stress distribution. In practice, thicker

adhesive layers cause a greater misalignment of the loads acting on the adhesive layer, creating important bending moments which greatly reduce the strength of the adhesive layer. Given this, one would be tempted to design a joint with an almost zero thickness to minimise bending moments, but this also results in poor joint performance, since very thin adhesive layers are highly susceptible to the appearance of areas where there is simply no adhesive, with the adherends directly in contact, greatly reducing the load-bearing capacity of the joint.

## 8.5.2. Effect of overlap length

#### 8.5.2.1. Overlap length and adhesive type

Figure 8.15 summarizes the combined effect of adhesive type and overlap length on the performance of an adhesive SLJ. We can first analyse the case of a rigid adhesive used to bond a high strength aluminium alloy. For very small overlap lengths, joint strength will be mainly limited by the strength of the adhesive layer, which occupies a small resistant area (bonded area). The Volkersen model is suitable for this case because the adhesive behaves elastically. With an increase of the resistant area, a linear increase in the strength of the joint is obtained, growing until a plateau is reached. From this point on, the performance of the joint can no longer be significantly improved with an increase of the overlap length.



Figure 8.15. Schematic representation of the evolution of the failure load as a function of the overlap length for two different types of adhesives.

This evolution is due to the presence of stress peaks at the ends of the overlap, the main cause of failure of rigid adhesive layers. These stress peaks will always be present even if very long overlaps are used, leading to a virtually unloaded central portion of the overlap.

In such cases, lengthening the overlap is a highly ineffective path to increase joint strength. Very ductile adhesives can yield before failure and redistribute stresses evenly along the entire overlap length. In this way, the formation of stress concentrations at the ends of the overlap is less likely and the adhesive can utilise all the available bonded area, as described in the Hart-Smith model. The failure load will be almost directly proportional to the overlap length allowing the generalised yielding model to be effective. An extremely elastic and flexible adhesive, such as a silicone or a polyurethane would also lead to the same results, since these properties are conducive to stress uniformity.

#### 8.5.2.2. Overlap length and adherend strength

The combined effect of adherend type and overlap length on the behaviour of an adhesive joint is summarized in Figure 8.16. If we bond a high strength, heat treated duraluminium alloy with a ductile adhesive, we will find a linear relationship between the failure force and the overlap length, exactly for the same the reasons we have described in the discussion of adhesive type. Similarly, this situation is suitable for use of the generalized adhesive yielding model.





If we consider a joint made using the same adhesive and adherends with low tensile strength, such as an untreated aluminium alloy, we will initially find a relatively linear increase in joint strength as we increase the overlap length, which will eventually stabilise at a plateau. However, this plateau is not the product of adhesive failure, but adherend yielding. As the low strength metallic adherend begins to yield, large stresses are imposed on the adhesive that force it to fail almost immediately. Therefore, further increasing the length of the overlap

will not produce any improvement as the strength of the adhesive is now the main limiting factor. This case is shown as a red line in Figure 8.16. Adherends with intermediate strength will also exhibit a plateau, but at a higher failure load value. This behaviour is represented by the blue line in Figure 8.16. Joint strength prediction for these cases can be accomplished using a model that considers the plastic deformation of the adherends (such as adherend yielding or Adams).

In sum, the strength of the adherend is a major factor in the response of a joint to variations overlap length. High strength adherends allow ductile adhesives to make use of the available overlap length, whereas low strength adherends limit joint performance, since they will eventually yield plastically and cause the adhesive layer to fail.

#### 8.5.2.3. Overlap length and composite adherends

We have previously discussed how peel stresses in the adhesive layer can be transferred to the composite adherends and cause interlaminar failure. In fact, this failure mode is also directly related to the overlap length. In joints with shorter overlap lengths, the peel stresses generated at the ends of the overlap are not sufficient to cause interlaminar failure of the composite adherends but for increased overlap lengths, the adhesive layer will eventually generate peel stress levels that can cause interlaminar failure of the composite adherend. In these cases, increases in the overlap length will no longer result in improved joint performance, leading again to a plateau of the failure load since the strength of the joint is *de facto* determined by the strength of the composite. This effect is shown in Figure 8.17.



Figure 8.17. Schematic representation of the evolution of the failure load as a function of the overlap length for composite joints bonded using two different types of adhesives.

The strength prediction of a composite thus requires a model that is able to calculate peel stresses, as is the case of the Goland and Reissner model or a finite element analysis.

#### 8.5.3. Presence of disbonds in the overlap

The presence of disbonds along the overlap length of an adhesive layer can also have a important effect on the performance of a bonded joint, although this effect is highly dependent on the adhesive type and the nature of the adherend, as shown in Figure 8.18. In a ductile adhesive, the failure load of the joint is quite sensitive to the size of the disbond and the type of adherend. Since a ductile adhesive can redistribute stresses over the full overlap length, the presence of a disbond in the centre of the overlap will reduce the load bearing capability of the joint, a case which is evident with the use of a high strength aluminium adherend. The adhesive global yielding criteria fits this case perfectly. Should the ductile adhesive be used to bond adherends of lower strength, the limit on joint performance will effectively be the yielding of the adherend, which is also practically unaffected by the presence of the disbonds. This case is, of course, properly modelled by the yielding of the adherend.

The scenario is quite different for a brittle adhesive where, in this case, the adhesive is not able to redistribute stresses and joint performance is controlled solely by the material located at the edges of the overlap. Thus, the presence of disbonds in the centre of the overlap does not drastically affect the joint performance. Even the change in adherend strength has a limited effect, since this type of adhesive cannot generate loads which are sufficient to induce adherend yielding.



Figure 8.18. Influence of disbonds on the performance of bonded joints.

## 8.5.4. Effect of service temperature and thermal stresses

The service temperature can have an important effect on the performance of a bonded joint, especially for joints which will operate in aerospace applications, including extremely high temperatures associated to frictional heating or engine components and very low temperature during flight at very high altitudes. Adhesives, as polymeric materials, are highly temperature sensitive and can provide insufficient cohesive strength if forced to operate above their glass transition temperature. Thus, when designing a joint that will see service in aeronautical applications, the mechanical properties used in the models must correspond to those found at the effective service temperature.

Residual thermal stresses are another important effect of temperature variations. Since adhesives and adherends have very different thermal expansion coefficients, these materials will expand very differently. However, due to the nature of the joint, it becomes physically impossible for this expansion to occur freely and this leads to the formation of residual stresses acting on the adhesive layer and the adherends. In practice, residual stresses are not highly damaging when the adherends are of the same material but joints with dissimilar adherend materials will experience severe thermal residual stresses even before the application of a mechanical load. This effect is schematically shown in Figure 8.19.



Figure 8.19. Residual stresses induced in the adhesive layer by the use of adherends with highly dissimilar coefficients of thermal expansion.

## 8.6. OPTIMIZATION OF THE BEHAVIOUR OF BONDED JOINTS

## 8.6.1. Use of adhesive fillets and adherend modifications

The introduction of a fillet represents a simple and highly effective method for the improvement of joint strength. A fillet will stem from the natural flow of uncured adhesive from edges of the adhesive layer and create a smooth connection between the adherends in a stress critical area. Fillets provide a smoother and more uniform shear stress distribution, allowing stress transfer over a larger area, leading to a joint behaviour that is closer to that of a ductile adhesive (Figure 8.20).



Figure 8.20. Influence of the presence of a fillet in the shear stress distribution of a bonded joint.

At its most basic, adhesive fillets do not even require additional effort to create them, since the excess adhesive will naturally produce a fillet shape that can transfer loads and lessen the stresses present in those regions. Nonetheless, it is possible to precisely control the shape of the fillet in order to improve its effectiveness.

The geometry of the adherends can also be modified to reduce the stress concentrations at the ends of the overlap length. There is a wide range of modifications that can be made to the specimens, such as the introduction of outer or inner chamfers. These changes can also be combined with changes in the adhesive layer for added effect, as illustrated in Figure 8.21.



Figure 8.21. Example of geometrical modifications made to the adherend and the adhesive layer to minimize stress concentration at the edges of the overlap.

#### 8.6.2. Mixed adhesive joints

Joints that combine two or more adhesives in the same adhesive layer are known as mixed adhesive joints (schematically shown in Figure 8.22), using their properties synergistically to achieve increased performance.



Figure 8.22. The concept of a mixed adhesive joint, manufactured using two different adhesives.

A mixed joint typically features a ductile adhesive at the ends of the overlap and a rigid adhesive in the central section of the joint, an area less subjected to large deformations during loading. The presence of the ductile adhesive at the ends of the overlap reduces the effect of stress concentration, while the rigid adhesive in the central part will support large loads without failure. A well-designed mixed adhesive joint should exhibit mechanical performance superior to that of the sum of its parts. Besides improving static joint strength, mixed adhesive joints also have the potential to improve the thermal behaviour of bonded joints, ensuring adequate performance under very large temperature ranges. This feature is especially useful for aerostructures of high-speed aircraft and bonded heat shield designs for spacecraft. The greatly expanded thermal range is achieved by combining high temperature resistant adhesives and low temperature resistant adhesives in a single joint (as seen in Figure 8.23). Fortuitously, this combination is perfectly well suited for the mixed adhesive joint concept, since the low temperature adhesives (such as silicones) are inherently flexible and the high temperature adhesives (such as temperature resistant epoxy formulations), are inherently rigid.



Figure 8.23. Schematic representation of the synergistic effect of combining two adhesives which respond differently to temperature in the same bonded joint.

## 8.6.3. Functionally graded joints

Graded joints can be seen as an idealized version of the mixed adhesive joint where instead of using a discrete approach, with distinct adhesives, there is now a smooth variation of the material properties. The working principle of a graded adhesive joint is show in Figure 8.24, showing how material properties gradually vary along the overlap.



Figure 8.24. The graded adhesive joint concept and a schematic representation of the desired material properties.

A graded joint is theoretically advantageous over a mixed adhesive joint since the adhesive properties match exactly what is required for each location of the joint. However, it is much more difficult to implement in practice and only recently has the production of fully graded joints been experimentally demonstrated and published. This was obtained by means of a differentiated curing process, using induction heating. The resultant functionally graded joint showed an improvement of ductility and high strength when compared with isothermally cured joints.

#### 8.6.4. Techniques for delamination avoidance in composites

As discussed above, the onset of delamination in composites is a major limiting factor on the performance of bonded joints, driven by the low resistance of composites to the transverse loads generated in the vicinity of the bonded joint.

The subject of composite failure driven by adhesive joining has been known for almost as long as composites have been used in structural applications in the aerospace sector. Initially, the only form of pre-preg available was unidirectional and this led to design solutions which made use of this unidirectionally to improve the strength of the joint, driven by experience. For example, when 90-degree plies were placed near the adhesive, it was quickly found that the that this led to cracking of the unsupported matrix (as shown in Figure 8.25). Thus, laminates suitable for bonding almost always included a ply of 0-degree orientation near the adhesive, ensuring that the stresses carried by the adhesive layer can be effectively transferred to the fibres (and not to the matrix).



Figure 8.25. Formation of cracks near the adhesive with the incorrect usage of a 90-degree composite ply adjacent to the adhesive.

As the use of bonded composite structures expanded, so did the techniques available to mitigate composite delamination. Currently, the most widely employed are the use of Z-pins, composite stitching, the adoption of fibre metal laminates and local and global toughening of composites.

The use of Z-pins allows to reinforce a composite through the thickness direction using metal or composite pins, which are usually inserted before the composite resin has cured. Z-pinning significantly increases of the composite strength under peel loads, resulting in an improvement of the resistance to delamination, although at the cost of a more complex manufacturing procedure. Stitching a composite, in the through the thickness direction, is based on the same concept of the z-pinning technique. The improvement of adhesive joint performance achieved using stitching follows the same mechanism that underlies the z-pinning process, as the stitches carry the transverse load between the adherends, leaving the adhesive and the resin of the composite relatively unloaded. These methodologies are shown schematically in Figure 8.26.



Figure 8.26. Thru-the-thickness reinforcement methods for composites using thread stitching and z-pinning.

Composites toughened with metals, known as fibre metal laminates (FML) are also extensively used in aeronautical applications. These consist of metallic layers interwoven with fibre reinforced polymers (as shown in Figure 8.27). The presence of the metallic layers provide added toughness and ductility to composite, creates pathways for stress redistribution in bonded joints with large peeling loads, thus being much less susceptible to delamination failure. However, it is crucial to ensure that there is a good level of adhesion between the

metallic and composite plies as otherwise the FML will not display any load bearing capability in the transverse direction.



Figure 8.27. Typical construction of a fibre metal laminate used in aerospace structures.

Finally, we can discuss local and global composite toughening processes. In local toughening, flexible and tough polymeric strips are strategically placed at the overlap ends and co-cured with the prepreg. This concept aims to precisely increase the toughness of the composite adherends just in the in areas where the stress concentrations are higher. In contrast, global toughening corresponds to composites which are coated with a fibre-reinforced high toughness resin, that is co-cured with a CFRP plate, resulting in a new composite with a stiff core but a tougher surface. This surface layer deals much better with the peel loads generated by at the bonded joints, distributing these loads over a much larger surface. Both cases are illustrated in Figure 8.28.



Figure 8.28. A globally toughened monolithic composite panel optimized for bonding, using a high toughness resin surface layer, reinforced with glass fibre fabric.

## 8.6.5. Hybrid bonded joints

Hybrid joints are those which combine adhesive bonding with other joining methods such as welding, riveting or bolting. An example of a hybrid joint that combines adhesive bonding with riveting is shown in Figure 8.29.



Rivet-bonded joint

Figure 8.29. Example of a hybrid joint, combining adhesive and riveting.

Hybrid joints have multiple uses. For example, they can be used to provide an increase joint performance but are also able to simplify the manufacturing process by providing an effective fixation method that holds the joint together while the adhesive hardens. In the aerospace industry, spot riveting and adhesive bonding are often found combined in primary structures, since riveting instantly ensures a solid connection between components. In service, both the riveted connection and the adhesive layer contribute to the strength of the airframe. Furthermore, hybrid joints also improve safety by increasing redundancy. Should one of these joining techniques fail, the other should still be able to provide cohesion to the structures, avoiding complete failure.



# DURABILITY

# 9. DURABILITY

As we have seen throughout this book, adhesive joints have been successfully used as an alternative to the traditional joining techniques in the manufacture of primary and secondary aircraft structures, growing in importance as the composite content in aircraft increased decade after decade. However, ensuring the durability of bonded joints is still highly challenging, especially when compared with other joining methods. Some design concepts, such as damage tolerance, have also had limited development, especially for use in load bearing structures such as the stringers bonded to the skin in airframes (fuselage, wing, and tail control surfaces) in civilian and military aircraft.

In light of these limitations, the application of bonded joints in the aerospace sector is still fundamentally limited to hybrid joint configurations or, if used alone, relegated to secondary structures, designed based on the fail-safe philosophy as mentioned in some regulations. Adhesives are also often used to bond crack stoppers to some sections of the airframe structures where access is limited in service. For these repairs, a fail-safe approach should always be followed, with the load acting on the fuselage being mainly transferred through the adhesive layer and the bonded patch. Figure 9.1 shows schematically a bonded patch used as a crack retarder in an airframe.



Figure 9.1. Adhesively bonded crack retarder in the airframe skin, located between stringers.

An increase in durability of the joints and precise estimations of the service life of the bonded structures can improve the maintenance programs and reduce operational costs. This is especially important since there is no NDT methodology method suitable for analysing all bonded joint configurations and the different defects that they can exhibit.

Several numerical and experimental techniques have been developed to achieve a precise life estimation and joints should pass fatigue and environmental testing, defining the service limits through an extensive experimental program. The amount of experimental data necessary to define the design load limits is much higher when composite adherends are used, a case which is becoming ever more common with the greater use of composite commercial aircraft. The current chapter introduces and describes varied methodologies for assessing the durability of adhesively bonded joints, divided into environmental effects and loading conditions.

## 9.1. ENVIRONMENTAL EFFECTS

## 9.1.1. Hygrothermal ageing

Airframes experience a wide range of environmental conditions during service but, for adhesive joints, one of the most aggressive conditions is a wet environment. In this condition, water will ingress into the adhesive and water molecules will bind with the adhesive chains leading to moisture induced ageing. Fluid molecules can also occupy the free space within the adhesive layer (Figure 9.2). These molecules can also penetrate through the interface of the adhesive and the adherend and degrade the interfacial properties. Adhesive ageing due to the exposure to a fluid can significantly reduce the durability of the bonded joints and the estimation of the service life of aged bonded joints is of paramount importance in the aviation sector. In general, water and hygrothermal ageing of adhesives have been extensively analysed, but only limited research exists on the ageing response of adhesives subjected to other fluids typically found in aircraft, such as fuels, hydraulic oils and de-icing fluids. As aeronautical structures experience a wide range of temperature during flight, and since there is a significant interaction between the ageing process and the environmental temperatures it is also important to characterize the mechanical properties of adhesives aged at different temperatures. Furthermore, one of the main issues associated to adhesive bonding in airframes is interfacial failure due to ageing, which requires the use of highly specific surface treatments and preparations. Many of these procedures are well defined in regulations, especially for the case of bonded repair patches.

Swelling, the increase of the volume of the adhesive that takes place as the fluid ingresses through the adhesive layers, can induce residual stresses within the adhesive layer which might lead to premature failure of the joint. Plasticization of the adhesive is also a product of ageing, which changes the durability and the mechanical performance of the bonded joints.



Figure 9.2. Schematic representation of the two different water absorption processes.

The determination of the rate of water/fluid diffusion in the adhesive layer is one of the key activities in durability analysis of aged bonded joints. For this process, the diffusion coefficient should be experimentally and numerically obtained. In the experimental component of this analysis, a bulk adhesive plate is manufactured, fully dried and immersed in the fluid. The weight of the plate is then measured at regular time intervals. The amount of fluid uptake as a function of time can be calculated by controlling the weight increase of an adhesive plate immersed in water. By dividing the amount of fluid uptake by the initial weight of the plate, the fractional mass uptake is obtained (known as  $M_p$ ). Plotting  $M_t$  as a function of time will fully characterize the fluid absorption process of the tested adhesive. Figure 9.3 shows a typical curve of the experimental fluid uptake as a function of time for an adhesive.



Figure 9.3. Fick's law curve and the experimental data point for an adhesive immersed in water.

The initial datapoints shown in Figure 9.3 and the level of fluid absorbed at saturation are used to define key parameters of the Fick's law. This law precisely defines how the fluid ingresses in adhesives exposed to a wet environment.

As discussed before, two different phenomena take place during water uptake. One of them is physical ageing. In physical ageing, the water/fluid molecules occupy the free spaces within the adhesive material. Should the joints be dried, the water/fluid molecules will evaporate and the adhesive properties, once degraded due to the ageing process, will now be restored. The desorption process is usually much faster than the absorption.

In contrast, damage generated during chemical ageing (or hydrolysis) is not recoverable after drying. In this case, the water/fluid molecules bind with the polymer chains as shown in Figure 9.2. The occurrence of chemical ageing can be ascertained by analysing the chemical structure of the adhesives during the ageing process.

The Fick's law parameters can also be determined for the desorption process. In this case, the weight of the test sample should be controlled at regular time intervals during the drying procedure in a dry environment (for example in a container filled with silica particles). However, since the desorption process is very fast, the time intervals should be much shorter than those in water absorption tests. It should be mentioned that the ageing response of the adhesive joints is highly dependent on the type of the ageing fluid. For example, for joints exposed to salt water or exposed to jet fuels the absorption process is much slower than that reported for distilled water (see Figure 9.4).



Time, t (s<sup>0.5</sup>)

Figure 9.4. Water absorption and desorption (drying) of an adhesive immersed in distilled water, salt water and fuel.

Furthermore, it is important to note that the rate of absorption is just one of the factors governing the ageing process of an adhesive. The sensitivity of the adhesive properties to
the level of ageing is also of paramount importance and must be fully understood for a successful adhesive selection process.

Typically, research on adhesive ageing has only considered single cycle ageing of adhesives, which means that only a single water uptake cycle is analysed. However, most bonded joints (such as those used in aircraft structures) are exposed to cyclic ageing conditions, where water absorption and desorption cycles are repeated. Using single cycle data to model these cases will overestimate the endurance limit of the bonded joints exposed to cyclic ageing conditions and a non-conservative prediction may lead to a premature catastrophic failure.

As discussed above, ageing is a multi-phenomenon process and can significantly reduce the durability of the bonded joints. Degradation of the adhesive properties, reducing the interfacial strength, swelling and inducing residual stresses within the adhesive layer (as shown in Figure 9.5), are a list of phenomena that take place during the ageing process.



Figure 9.5. Schematic representation of the effect of swelling stresses on the stress level in adhesive joints.

To analyse the durability of the aged samples, tests should be conducted on joints subjected to different levels of ageing. Using the obtained results, the effects of ageing level on the strength of the bonded structures can be precisely calculated (see Figure 9.6).



Figure 9.6. Effect of hygrothermal ageing on the tensile strength of adhesives.

#### 9.1.2. Temperature

Adhesives used in aerospace applications usually experience a very wide range of temperatures during every single flight, which can induce residual stresses within the adhesive layer especially in case of dissimilar joints which are quite common in aeronautical applications. On the other hand, the mobility of the polymer chains increases at higher temperatures, which further reduces the performance of the joint (see Figure 9.7). As described for the ageing conditions, different characterization processes should be conducted to evaluate the durability of bonded joints as a function of the testing temperature. These tests are mainly based on the standard material characterization tests but carried out inside temperature-controlled chambers.



Figure 9.7. Effect of temperature on the stress-strain response of adhesives.

#### 9.1.3. Radiation damage

Radiation is, of course, known to be harmful to human life, but sources of this highly energetic, ionizing radiation can also damage aerospace structures. In addition, spacecraft are highly exposed to radiation, as they operate in an environment which does not benefit from the radiation shielding provided by the atmosphere. Bonded joints can be damaged by this radiation, which can lead to a degradation of adhesives' properties and reduce their durability, potentially leading to a catastrophic structural failure.

The effects of radiation on adhesive properties have been the target of study in recent decades and, as shown schematically in Figure 9.8, chain crosslinking and chain scission were found to occur as the result of this exposure. In some cases, a combination of these two main mechanisms can also take place simultaneously.



Figure 9.8. Effects of radiation on an adhesive.

The precise design of the component and the joint itself must consider the effects of radiation, for example, maximizing shielding of the adhesive layer. However, it should be noted that the response of adhesives to radiation can be varied. Consequently, a general statement about the effects of radiation will never be valid for all joint configurations. In some specific cases, radiation can even lead to joint strengthening, as it will further crosslink and harden an adhesive. Thus, conducting radiation exposure experiments is often indispensable for some bonded structures used in the aeronautical industry, although this is a very costly process. Due to their simplicity, single lap joints are often used for analysing the response of adhesives exposed to a radiant environment. The performance of these joints can be analysed both in terms of static strength and fatigue life.

Several parameters alter the response of adhesives exposed to radiation environments. For example, the atmosphere where radiation durability tests are conducted can significantly affect the results and the ambient temperature also has a significant influence on the intensity of radiation-induced damage. Even the presence of special additives can change the dose of radiation absorbed by the adhesive layer in a bonded joint.

### 9.2. LOADING CONDITIONS

#### 9.2.1. Fatigue

In an airframe subjected to flight cycles, aerodynamic loads and vibrations, bonded joints are continuously subjected to repetitive and cyclic loadings. One of the key sources of this alternating stress is the internal pressurization of the cabin, which creates significant hoop stresses, as illustrated in Figure 9.9.



Figure 9.9. Stresses generated in an aircraft fuselage due to the pressurization cycles.

Although the design ultimate load is usually considered as one of the main certification requirements for bonded joints in the aeronautical industry, it is not enough to meet the durability requirements under fatigue loads, even if no crack grows during the quasi-static design ultimate load test. In Figure 9.10, the most typical fatigue loading conditions are shown. Although fatigue damage models and test procedures are based on a constant amplitude fatigue loading, in reality joints are subjected to a random variable amplitude loading, more closely resembling the waveform schematically shown in Figure 9.11.

Fatigue failure is a complex mechanism as is affected by several parameters including the loading conditions. The maximum load, loading range ( $\Delta \sigma$ ), average load ( $\sigma_m$ ), and the loading amplitude ( $\sigma_a$ ) are the most important fatigue loading parameters that can change the fatigue durability of a joint. R ratio defined as the ratio of the minimum load to the maximum load is also an important parameter. Furthermore, a key parameter that is rarely included in durability analysis models but can significantly change the durability of the bonded joints is the loading frequency. Accordingly, it is recommended to conduct the durability tests at frequencies consistent with that of service loads. Thus, the simplified sinusoidal constant amplitude cyclic load shown in Figure 9.10 may not always lead to a precise result and it is important to consider more complex loading conditions, usually of variable amplitude as schematically shown in Figure 9.11.



Figure 9.10. A typical sinusoidal fatigue loading.

Equations 9.1 to 9.4 list the main parameters of a sinusoidal fatigue loading curve.

$$\Delta \sigma = \sigma_{\max} - \sigma_{\min} \qquad \qquad 9.1.$$

$$\sigma_a = \frac{\sigma_{\max} - \sigma_{\min}}{2}$$
 9.2.

$$\sigma_m = \frac{\sigma_{\max} + \sigma_{\min}}{2}$$
 **9.3.**

$$R = \frac{\sigma_{\min}}{\sigma_{\max}} \qquad 9.4.$$



Figure 9.11. Schematic of a variable amplitude fatigue loading.

Fatigue is a time dependent damage mechanism, where the properties of the adhesive degrade cycle by cycle and will finally lead to a failure of the joint. Although the applied load is much lower than the static strength of the adhesive, even low scale fatigue loads can significantly reduce the span life of a joint.

The total fatigue life of a joint can be divided into two stages. The first of these stages corresponds to the fatigue initiation life. In an undamaged joint, a crack will eventually initiate after a sufficient number of load cycles has accumulated. This number of cycles sustained before crack initiation is known as the fatigue initiation life. For some materials, and depending on the joint geometry, the fatigue initiation life can be quite short while for some others it can be much larger, representing the majority of the total fatigue life the joint. The second stage of the total fatigue life corresponds to the propagation of fatigue crack. For some materials the crack can propagate very fast while in some other cases the

fatigue crack growth is very slow. To analyse the durability of an adhesive joint under fatigue conditions, it is important to first know what are the fractions of the fatigue initiation and fatigue crack propagation phases, relative to the total fatigue life of a joint. Accordingly, a total fatigue life approach or a fatigue crack growth model should then be employed. Both approaches are discussed in the following sections.

#### 9.2.1.1. Total fatigue life (S-N) approach

A durability analysis based on the total fatigue life (S-N) methods means that the joint will be subjected to fatigue loads until its failure and it is assumed that the crack propagation is very fast. The applied fatigue load and the corresponding fatigue life are used to analyse the joints. In the S-N technique, the obtained fatigue life is plotted against an equivalent stress parameter. The equivalent stress parameter is a composition of stress components. Various alternatives to this stress component composition have been proposed in the literature. So far, several different relations have been proposed for the equivalent stress. A good equivalent stress should be able to collapse all the S-N curves obtained for different loading conditions into a single curve which is called *master curve*. To obtain the master curve, an adhesive should be characterized in terms of fatigue at different mode mixities and load levels. This is achieved using standardised specimen geometries, such as Arcan joints, which are manufactured, and fatigue tested at different loading angles.

The S-N method usually requires a simple linear elastic analysis to be conducted on the joint geometry under analysis and the defined equivalent stress should be measured within the bondline. By knowing the effective stress value, the corresponding life can be estimated using the previously determined master curve. Figure 9.12 schematically shows the procedure used to construct a master curve for adhesive joints. Although simple, the use of the master fatigue curve method has some drawbacks. First, it is quite sensitive to the mesh size used in the numerical simulation. Second, the equivalent stress is actually not a general stress component and may not be used for all joint geometries. Third, defining the point where the equivalent stress should be measured is still challenging and can be somewhat arbitrary.

Due to these limitations, a secondary strategy is usually preferred by designers, called the direct method. In the direct method the final, practical joint geometry is directly subjected to the load cycles. No characterization is normally needed since the obtained fatigue life is for the real joint . No post processing is usually needed and the obtained results can be directly used for design purposes. However, this strategy is very costly and the results cannot be used to evaluate the fatigue durability of other types of joints. For some structures, a sub-component level durability analysis is considered since testing the real structure in its full scale is often not possible. However, in some applications the service loads are biaxial or multiaxial in nature, making them difficult to apply. Accordingly, the first method (indirect approach) is often chosen for design purposes since it is more general in nature.



Figure 9.12. Schematic view of the S-N procedure for fatigue life estimation of bonded joints.

The S-N response of the adhesive is also significantly influenced by environmental conditions. Fatigue degradation can be accelerated if the bonded joints are subjected to an aggressive environment, such as exposure to fuel, hydraulic fluid, water, and high or low temperatures. Figure 9.13 schematically shows the effects of different parameters on the S-N response of an adhesive. Constructing a master curve by taking all these effects into account is, of course, a challenging prospect, requiring many characterization tests under varied conditions. Accordingly, the direct method is often more practical for the design of joints subjected to harsh environmental conditions when in service.



Figure 9.13. Effect of temperature and ageing on the S-N curve of adhesives.

#### 9.2.1.2. Fatigue crack growth approach

Although the S-N method is simple and precise enough to use with most components, lightweight aerostructures are mainly designed based on the damage tolerance concepts, requiring fatigue crack growth to be analysed in parallel with the total fatigue life. Fatigue crack growth analysis helps to design an appropriate fracture control plan for different sections of an airframe. Fatigue crack growth analysis is of paramount importance in cases where the crack growth forms the main part of the fatigue life the joint. DCB, end notched flexure (ENF) and mixed mode bending (MMB) tests are used to characterize the adhesive in mode I, mode II and mixed mode, respectively. Then, the rate of increase in crack length (da) as a function of load cycles (dN) is plotted against an equivalent fracture energy parameter. The ratio of da/dN is called fatigue crack growth rate. By plotting the da/dN as a function of fracture energy in a log-log diagram, a curve, called the Paris law, is obtained. Depending on the rate of crack growth, the Paris law curve can be divided into three sections, as shown in Figure 9.14.



Figure 9.14. Paris law curve and its three main stages.

The first region corresponds to the threshold condition where the strain energy release rate is below a threshold value. In this condition the rate of crack growth is negligible. However, as soon as the energy reaches the  $G_{th}$ , the crack starts to propagate. In this region, there is almost a linear relation between the rate of crack growth and the strain energy release rate. This is the second stage of the Paris law. The slope of the curve in this region is almost constant and called *m*. Eventually the crack size reaches a critical value and the rate of crack growth is no longer stable, leading to an exponential growth. This the most critical region of the Paris law curve which must be avoided at all costs with proper joint design. For the linear part of the Paris law a relation can be defined as follows:

$$\frac{da}{dN} = C(G)^m$$
 9.5.

where *C* and *m* are Paris law constants and should be obtained experimentally, as shown in Figure 9.14. Based on the fatigue crack growth strategy, the fatigue life is equal to the number of cycles required to increase an initial flaw size of  $a_0$  to a critical size ( $a_c$ ). By knowing the values of  $a_0$  and  $a_c$  and by considering the slope of the Paris law curve, one can reliably estimate the fatigue life of the joint.

Environmental and loading conditions can also significantly change the slope of the Paris law curve. Figure 9.15 schematically shows the effects of different parameters on the fatigue crack growth behaviour of an adhesive.



Figure 9.15. Effects of mode mixity, temperature and humidity on the Paris law curve.

#### 9.2.2. Creep

Another time dependent phenomenon that influences the service life of the bonded joints is creep. Creep is defined as the deformation of an adhesive or joint subjected to a constant load for a given period of time. In some cases, creep deformation can eventually lead to joint failure. Creep takes place in three stages as shown in Figure 9.16. During the first stage, the creep rate is initially quite high but rapidly decreases over time. This step is called the transient stage. After a specific time has passed, the creep behaviour transitions into a longer second stage where creep rate is generally constant. Creep damage within the adhesive layer increases with time and as soon as the damage reaches a critical value, the final accelerating phase starts, whereupon the creep deformation exponentially increases with time in an unstable manner. This behaviour continues until the joint eventually fails.



Figure 9.16. A typical creep curve and its three main stages.

Creep is known to be strongly influenced by the level of the applied stress and by the service temperature. The moisture level can also affect the rate of creep deformation, although with a more limited effect. Figure 9.17 typically shows the creep response of an adhesive as a function of different environmental and loading conditions.





In general, the service loads acting on bonded joints are generally much lower than the static strength of the joint but the service life is long enough to cause creep failure in the joints. Even in cases where applied load is of a cyclic nature, we can be faced with load condition where the average fatigue load is also effectively acting as a creep load. To avoid a catastrophic failure of an airframe structure, for example, time intervals between the inspection should be short enough to stop the bonded joints from reaching the third stage of creep deformation unchecked.

Equations that relate the applied adhesive stress to the corresponding displacement (strain) describe the constitutive law of the material and by knowing these relations, one can estimate the creep response of the bonded structure via diverse numerical approaches such as FEM. Different constitutive laws have been proposed by researchers to model creep. Almost universally, these models consider the viscoelastic response of the adhesive, exhibiting a deformation increase within the adhesive material even under unchanged static loads. These models are usually able to consider the ambient temperature as well, since the higher the temperate the higher the deformation rate at a specific creep time.

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