Mechanical properties of a silicone for high temperature applications

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Final project in MIEM

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Abstract

An increased use of adhesively bonded joints in industrial applications has renewed the interest of high temperature research in adhesive joints. This study aims to present the mechanical properties of a silicone for high temperature applications, especially in aerospace industry. To do this, in the experimental work, a RTV 106 silicone was used, a high temperature acetoxy adhesive sealant supplied by GE Bayer Silicones. The following tests were carried out: Thick adherend shear test (TAST), Single lap joint test (SLJ), and the Double cantilever beam test (DCB). This work consisted on making the specimens, testing them, and interpret the mechanical properties obtained.

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LIST OF ABBREVIATIONS

ADCB	Asymmetric Double Cantilever Beam
ASTM	American Society of Testing and Materials
CLS	Crack Lap Shear
DCB	Double Cantilever Beam
ENF	Edge Notched Flexure
FEUP	Faculty of Engineering of the University of Porto
ISO	International Organization for Standardization
LEFM	Linear elastic fracture mechanic
MMF	Mixed – Mode Flexure
PSA	Pressure sensitive adhesives
PVA	Polyvinyl acetate
SLJ	Single Lap Joint
TAST	Thick Adherend Shear Test
TDCB	Tapered Double Cantilever Beam

LIST OF SYMBOLS

a - crack length

a_0 - initial crack length

$\tilde{a}$ - debond length correction

b - width of specimen

c_g - lever length to center of gravity

C - compliance

E - Young`s modulus of adherend

G - strain energy release rate

G_c - critical strain energy release

G_{1c} - fracture toughness (from load to start of crack)

G_{1a} - fracture toughness (from arrest load)

h - thickness of the adherend, normal to plane of bonding

K_I - mode I stress intensity factor

K_{II} - mode II stress intensity factor

K_{III} - mode III stress intensity factor

K - stress intensity factor

K_c - critical stress intensity factor

P_c - critical load

GLOSSARY

Activators: Chemicals which can be applied directly to a surface, substrate or mixed with an adhesive to speed up the solidification of an adhesive.

Adhesion: The bonding forces between two different materials.

Adhesive: A material employed to attach one solid to another so that the two solids may function as a single unit.

Adhesive Failure: Loss of adhesion between the adhesive and substrate. The adhesive pulls cleanly away from the substrate.

Bond: The union of materials by adhesives.

Bond Line: The space or gap between two substrates which contain the adhesive.

Bond Strength: The amount of force a bond can sustain. Measured in Pounds per Square Inch (PSI).

Cohesion: The state in which the particles of a single substance are held together by primary or secondary valence forces.

Cohesive Failure: Loss of adhesion as a result of the adhesive rupturing, leaving adhesive on both substrates involved in the bond.

Composite: A material which is composed of two or more different substances.

Cure: To change the physical properties of a material by chemical reaction through condensation, polymerization or vulcanization. Usually accomplished by the action of heat and catalysts, alone or in combination with or without pressure.

Cyanoacrylate: A one-part adhesive that cures instantly on contact with mated surfaces. High strength, excellent adhesion to a wide variety of substrates, especially plastics. Poor shock resistance super glue.

Durometer: A device used to determine the hardness of a material.

Elastomer: A synthetic rubber, plastic or other polymer which can be stretched to at least twice its original length then return to its original shape with force.

Epoxy: A two-component adhesive with high strength and low shrinkage during cure. Epoxies are tough and known for their resistance to chemical and environmental damage, as well as their usefulness as structural adhesives. Some formulations are used as potting agents, while others are useful as thermal or electrical conductors.

Gel: A description of an adhesive as it hardens from liquid to solid. More often a solid than a liquid.

Lap Shear: Shear stress acting on an overlapping joint.

Mold: The cavity or matrix into or on which the plastic composition is placed and from which it takes its form.

Peel Strength: The amount of force required to peel a material off a substrate.

Polymer: A complex chemical compound made of similar compounds linked together (e.g. acrylics, epoxies, silicones, urethanes).

Rheology: The study of the flow of matter, especially the non-Newtonian flow of liquids and plastic solids.

Room Temperature: 70°F/21°C

Shear: The effect of forces acting in opposite but parallel directions.

Shear Strength: The maximum shearing force, per unit area, an adhesive bond will endure before failure.

Silicone: Any member of a family of polymeric products whose molecular backbone is made up of alternating silicon and oxygen atoms and which has pendant hydrocarbon groups attached to the silicon atoms. Used primarily as a sealant, silicone is known for its ability to withstand large variations in temperature (-38°C to +316°C). Silicone is reliable and is relatively easy to handle.

Substrate: Materials to be bonded together.

Tensile Strength: The maximum stress a material subjected to stretching can withstand without tearing.

Chapter 1

1. Introduction to adhesive and sealants bonding

1.1 Definitions and introductory notions

This study aims to present a technique for modern assemblies. Even if the adhesives have been used for thousands of years, due to the development of modern computing and experimental techniques, now it is possible to study their behavior in different applications before manufacture. Because this type of assembly has become increasingly important in industry there is now a better understanding of the phenomenon of adhesion.

Along with the transition from conventional materials like glass, stone, wood, and metal to lighter and more economical petroleum-based plastics, the adhesives and sealants industry has made much progress since these non-conventional materials need new methods of assembly and suitable bonding components. The global market for adhesives and sealants should reach $46.4 billion by 2012 [1]. Adhesives and sealants are two groups of formulated products that are chemically similar but functionally different.

An adhesive is a compound in a liquid or semi-liquid state that adheres or bonds items together. The type of materials that can be bonded using adhesives is virtually limitless, but they are especially useful for bonding thin materials. Adhesives usually require a controlled temperature.

A sealant is a viscous material that changes state to become solid, once applied, and is used to prevent the penetration of air, gas, noise, dust, fire, smoke or liquid from one location through a barrier into another. The main difference between adhesives and sealants is that sealants typically have lower strength and higher elongation than do adhesives. The universal definitions of adhesive is “a substance capable of holding at least two surfaces together in a strong and permanent manner” ,while the sealant is defined as “a substance capable of attaching to at least two surfaces, thereby, filling the space between them to provide a barrier or protective coating [2].

There are many advantages in using adhesives compared to other fastening methods, but there are some limitations, too as listed in the Table 1 [3].

The strength of an adhesive joint depends on the strength of the attachment of the adhesive to the material surface (substrate) termed adhesion and strength of the adhesive termed cohesion.

Adhesion is the attraction of two different substances resulting from intermolecular forces between the substances that are distinctly different from cohesion, which involves only the intermolecular attractive forces within a single substance. Adhesive or sealant joints may fail either adhesively or cohesively. Adhesive failure is failure at the interface between adherend and the adhesive.

Any assembly with adhesives comprises the following components:

- Two substrates
- The adhesive
- The interphase region; this interphase region is a thin region near the point of adhesive - adherend contact.

Table 1 Advantages and disadvantages of adhesives [3]

Advantages	***Disadvantage***
Fatigue - There are few, if any, stress concentrations associated with adhesive joints	Certain adhesives are vulnerable to attack by chemicals /water
Surface Area - For a lap joint, the stress is distributed over a relatively large area. This provides a strong joint with lower specific stresses	For the best joints surface preparation is important.
An adhesive joint can provide a sealing function in addition to a fastening function	Some adhesives require time to achieve design strength
For a high production process an adhesive joint can be extremely economical	An adhesive joint is intuitively not considered a sound joint
Compared to welding, there are no high temperatures required	Many adhesives have a temperature limitation
Adhesives are available for attaching a wide variety of different materials.	For mass production, reliable joints jigs and fixtures are important
Compared to bolted joints and rivetted joints there is less need for machined holes, and additional machined components	Many adhesives are toxic and require care when being applied.
The is no weight penalty associated with adhesive joints and the stresses developed in an adhesive joint are almost entirely imposed by the operating conditions	Adhesive joints are often vulnerable to localized high stress due to peeling effect

Depending on for their holding and bonding power the adhesives can be: structural adhesives and non – structural adhesives. In the first case, the strength of the adhesive is an essential parameter of the assembly, while non-structural adhesives have a much lower strength and performance.

The adhesion is the property behind the operation of any adhesive and sealants. Chemical adhesion is when two materials may form a compound at the joint. The strongest joints are where atoms of the two materials swap (ionic bonding) or share (covalent bonding) outer electrons. The science of adhesion requires the adaptation of multiple disciplines:

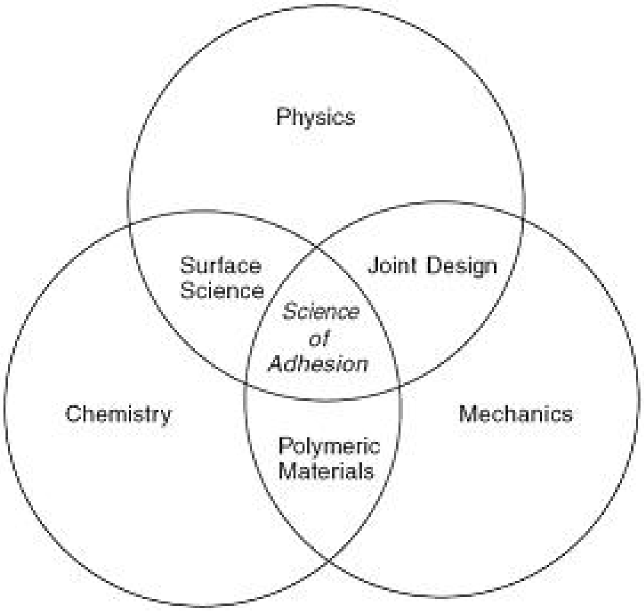


Figure 1 - Science of adhesion; Multidisciplinary graph [2].

1.2 Classification of adhesives

1. General classification of adhesives by structure

This category includes the following types of adhesives:

A) ***Thermosetting adhesives*** - Most of thermosetting adhesives are based on epoxies, polyesters, polyimides and phenolics.

B) ***Thermoplastic adhesives*** - Polyamides, cyanoacrylates, polyacrylates, polyvinyl acetate (PVA) are typical thermoplastic adhesives.

C) ***Elastomeric adhesives*** - Typical elastomeric adhesives are based on natural rubbers, silicones, acrylonitride butadiene (nitrile), neoprene, butyl, polyurethane, styrene-butadiene.

2. Classification of adhesives by curing method

A.) One-part adhesives

- **Heat activated curing adhesives.** Adhesives of this type (epoxies, urethanes, polyimides) consist of a ready mixture of two components.
- **Light/UV activated curing adhesives (acrylics, cyanoacrylates, urethanes).** Light activated adhesives are cured under a visible or UV light of appropriate wave length. Adhesives of this type usually contain photoinitiators enhancing curing reaction.
- **Moisture activated curing adhesives (RTV silicones, cyanoacrylates, urethanes)**.These adhesives are cured when they react with the moisture present on the substrate surface or in the air.
- **Anaerobics** acrylic-based adhesives cured between metallic substrates, surfaces of which are deprived of oxygen.
- **Pressure sensitive adhesives (PSA)** Adhesives of this type do not cure. Adhesive bonding forms as a result of a pressure applied to the substrates. The adhesion strength is determined by the pressure applied to the substrates. Typical example of pressure sensitive adhesive is self-stick tape.

B.) Two-part adhesives (epoxies, urethane, acrylics, silicones).

A two-part adhesive is cured when its two (or more) components are mixed. The components react chemically forming cross-links of the polymer molecules.

3. *Classification of adhesives by origin*

a.) Synthetic adhesives

Typical synthetic adhesives are epoxies, polyurethanes, cyanoacrylates, polyimides, silicones, acrylics, polyamides, cyanoacrylates, polyacrylates, polyvinyl acetate (PVA), nitrile, neoprene.

b.) Natural adhesives (glues)

- Animal glue.
- Casein
- Fish glue
- Vegetable-based glues (tapioca paste, soybean glue, starch glue)
- Natural rubber (latex) glue

1.3 Sealant Classification

The main difference between adhesives and sealants is that sealants typically have lower strength and higher elongation than do adhesives. Sealants fall between higher-strength adhesives at one end and extremely low-strength putties and caulks at the other. The large number and diversity of sealant formulations make their classification difficult.

Still the joints sealant can be broken down into six primary categories:

1. Acrylics

- Used in residential and light commercial construction, mainly for exterior applications
- Generally meet ± 12.5% movement (ASTM C 920, Class 12-1/2)
- May need special handling for flammability and regulatory compliance
- Can be painted
- Short open time; difficult to tool
- Exhibit some shrinkage upon cure
- Often used for perimeter sealing and low movement joints

2. Butyls (solvent – based)

- Excellent adhesion to most substrates
- Excellent water vapour transmission resistance
- Limited movement capabilities, generally up to ± 10%
- Excellent weathering
- Sometimes used in curtain wall applications where adhesion to rubber compounds is needed
- Most are stringy and difficult to apply neatly
- May show some shrinkage after cure; may harden and crack over time on exposed surfaces
- Some are not suitable for application where exposed to UV

- Some are non-hardening and are suitable for concealed application where sealant needs to remain pliable

3. Latex (water – based and including EVA)

- Used mainly in residential and light commercial construction applications
- Interior and exterior use
- Premium products meet ± 25% movement (ASTM C 920, Class 25)
- Excellent paintability (with latex paints)
- Very good exterior durability
- Exhibit some shrinkage after cure
- Not used for exterior applications, particularly on high rise construction, for applications undergoing significant cyclic movement, or for high-profile structures

4. Polyurethanes

- Used in industrial and commercial applications
- Very good movement capabilities, up to ± 50% (ASTM C 920, Class 25 and 50).
- Not used in SSG applications (avoid direct contact to glass)
- Excellent bonding, generally without a primer for many surfaces
- Can be formulated for good UV resistance, which may be compromised by a selected color
- Paintable
- Some formulations may contain low levels of solvent

5.Polysulfides

- First “high performance” sealant; mainly used in industrial applications (ASTM C920, Class 12-1/2 or 25)

- Poor recover limits their use in joints with high cyclic movements
- Can be formulated for excellent chemical resistance (especially for aviation fuel)
- Good performance in submerged applications
- Require a primer on almost all substrates

6. Silicones

- Structural bonding and structural sealant glazing (SSG) of glass to frames
- Excellent joint movement capabilities; can exceed ± 50% (ASTM C 920, Class 50 and Class 100/50)
- Excellent low temperature movement capability
- Excellent UV and heat stability
- Good adhesion for many substrates especially glass; a primer is recommended on certain substrates, particularly porous substrates
- Not paintable
- Used in protective glazing systems and insulating glass units to improve thermal performance (reduce heat loss). Also used for missile impact and bomb blast situations
- Acetoxy chemistry based sealants have strong odor, but newer chemistries have very low odor
- Adhesion, as for all sealant types, is adversely affected by less than perfect application conditions
- High, medium and low modulus sealants available
- May stain some types of natural stone without primers. Low staining potential formulations are available

1.4 Joint Design

Joint design is critical to the successful use of adhesives. Because of the low cohesive (as opposed to interfacial strengths) of adhesives, purely tensile joints must be avoided. When designing an adhesively bonded joint, it is not advisable to assume that the joint design should be the same as for traditional fixing methods like welding and brazing, it is better the operating load is transferred as a shear stress as opposed to a tensile stress. The design of the adhesive joint will play a significant factor in determining how it will survive service loads.

External loads produce local stresses that may be many times the average stress. These stress concentrations are often unexpected, and they may determine the actual force that the joint can sustain.

Typical forces, which may be applied to adhesive joints, are presented in the figure below [4]:

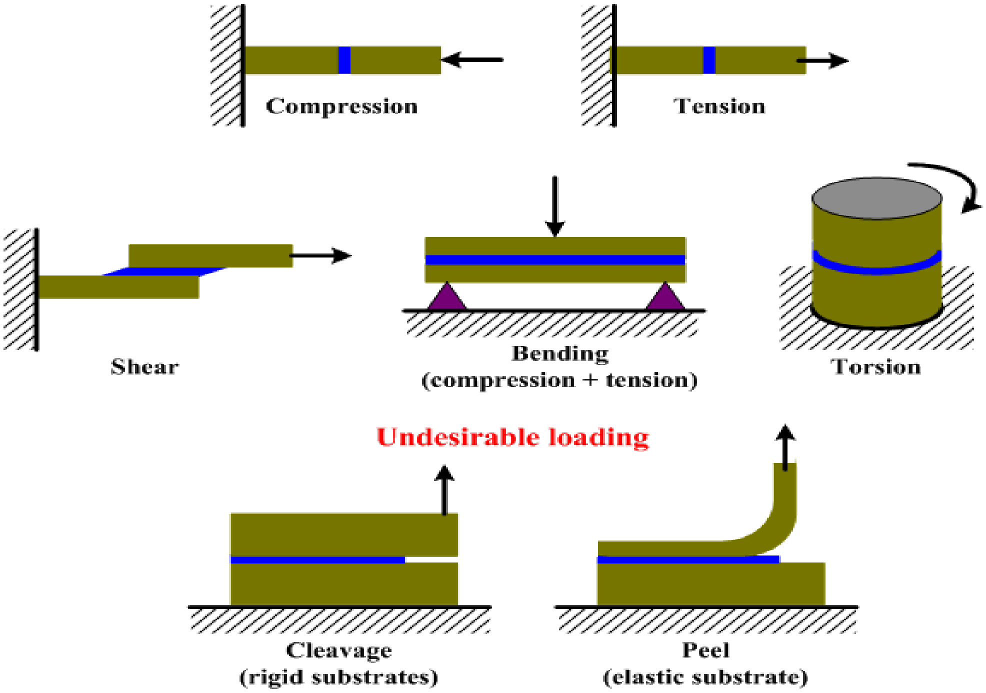


Figure 2 – Typical forces in adhesive joints [4].

When designing the joint it is necessary to:

- maximise tension, shear and compression forces;
- minimise peel and cleavage forces;
- optimise the area over which the load is distributed

The most typical design of adhesive joints are presented in Figure 3.

(a) Simple Lap Joint

(b) Tapered Lap Joint

(c) Rebated Lap Joint

(d) Single Strap Joint

(e) Double Strap Joint

(f) Double Strap Joint

Figure 3 - Typical design of adhesive joints [5].

In a simple lap joint made from thin metal sheet there are two sorts of stress: shear and peel as shown in Figure 4.

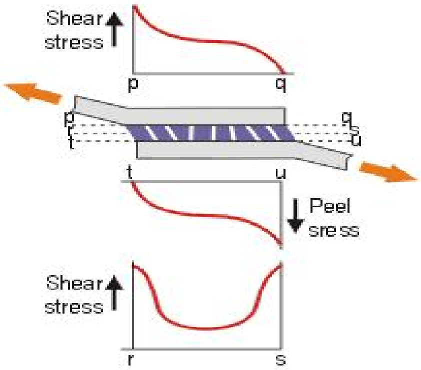


Figure 4 - Stress in a simple lap joint [6].

Shear stress results when forces acting in the plane of the adhesive try to separate the adherends.

Peel stress is defined as the force that pulls an adhesive apart by separating one flexible surface and one rigid surface.

Non-uniform stresses within the adhesive joint can significantly reduce the maximum strength of the joint. Non-uniform stress distributions generally cannot be eliminated, but they can be reduced through proper joint design and selection of certain design variables. There are a large number of variables affecting stress distribution, even in the most common joint designs. The following are most important:

1. Adhesive material properties
2. Adhesive thickness
3. Adherend properties
4. Geometry of the bond area

1.5 Adhesive properties

In the most structural application there are used the following types of adhesives: epoxy, silicones, cyanoacrylates, anaerobics, acrylics, polyurethanes, polyiamides, phenolics,bismaleimides. Their main properties of this adhesive are grouped in the Table 2 [7]:

Table 2 Adhesive properties [7]

ADHESIVE	PROPERTIES	SERVICE TEMPERATURE (ºC)	CURE	OBSERVATIONS
Epoxy	High strength Good solvent High temperature resistance Low cost Wide range of formulations	- 50 to + 121	One-part epoxies cure whit temperature Two-part epoxies cure at room temperature.	Impact resistance is poor, flammabi-lity is low and whit a moderate toxicity
Acrylics	Good peel and shear strengths Will bond dirty (oily) surfaces Moderate cost	- 73 to + 121	Room temperature cure	Good solvent resi--stance, 0,030 gap limitation, in max.
Cyanoacrylates	Fast bonding capability to plastic Long pot life Good adhesion to metal Poor resistance to moisture	- 30 to + 78	Rapid room cure (min.)	2700 tensile shear
Anaerobics	Good solvent resistance Nontoxic Indefinite pot life Moderate cost	- 55 to +149	Rapid room temp. cure in the absence of air or oxygen	Limited gap cure
Polyurethanes	Good flexibility at low temperature Tough Impact resistance Durability Short pot life	- 200 to + 79	Varying cure times	Fair moisture resistance 80 T-peel, piw

Silicones	Excellent sealant for low stress applications, high temperature resistance	- 60 to + 350	Room temperature	Low strength
Phenolics	Good strength rete-ntion for short periods of time	- 40 to + 175	Cure whit temperature and high pressure	Limited resistance to thermal shocks
Bismaleimides	Too rigid	- 50 to + 200	Cure whit temperature and high pressure	Low peel proper-ties
Polyimides	Thermal stability, dependent on a number of factors	- 40 to + 250	Cure whit temperature and high pressure	Difficult processability

High-temperature adhesives

The adhesive which needs to exhibit high-temperature capabilities has to maintain its mechanical properties at the intended service temperature and to maintain its structural integrity (resist thermal breakdown at elevated temperature). The former is controlled primarily by the glass transition temperature, Tg, while the structural integrity is largely dependent upon the inherent thermal stability of the polymer from which the adhesive system is produced. For short-term applications, that is, short durations at elevated temperature, the first requirement is of overriding importance, the latter can be, to a large degree, ignored. However, for longer-term applications, both requirements must be considered important.

There has been a growing requirement recently, particularly in the aerospace industry, for adhesives to withstand temperatures in excess of 150ºC for both short and long term applications. Epoxy resins, which currently form the basis of most structural adhesive systems, are generally formulated to yield high temperature capabilities of approximately 50–

100ºC. Although modest improvements in this capability are possible by, for example, variation in resin/curative functionality, together with the use of certain filler materials, 150ºC is generally considered the maximum permissible working temperature. Advantages of epoxy-based systems include relatively low cure temperatures, no volatiles are formed during cure, low cost, and a variety of available formulating and application possibilities. The higher temperature adhesives lose many of these advantages in favour of improved thermal aging characteristics.

The traditional structural adhesives capable of operating at temperatures in excess of 150 ºC for both short and long term applications can be divided into three classes, namely: phenolics, polybenzimidazoles and condensation polyimides [8].

Silicone adhesives

Silicones are used in numerous products, applications, and processes across all industries. These polymeric materials combine organic and inorganic character and possess unique physical, chemical, and mechanical properties that are unmatched by any other polymeric materials. Their success in a multitude of technologies is due to a range of properties that include low surface energy, hydrophobicity, chemical resistance, electrical insulation, resistance to weathering, stability to extreme temperatures, resistance to thermal shocks, high elasticity, good tear strengths, capability to seal or bond materials of various natures [9].

Silicones are classified as fluids, elastomers, and resins. Thus, silicone polymers may be fluid, gel, elastomeric, or rigid in their form. Good physical properties are maintained up to 260 ºC (short term up to 300 ºC or 350 ºC).

The silicone containing thermoset adhesives cure from the moisture in the air or adsorbed moisture from the surface being joined. They form low strength structural joints and are usually selected when considerable expansion and contraction is expected in the joint, flexibility is required (as in sheet metal parts), or good gasket or sealing properties are necessary. These adhesives can be used to join metals, glass, paper, plastics, and rubbers. However, long cure times, low strength and relatively expensive cost have limited their use.

1.6 The effects of environment on adhesive bonded

The effect of environmental factors on adhesively bonded joints plays an important role in the performance or durability of adhesives systems during their service life. Environmental factors that affect stresses include temperature and moisture. Another important factor is represented by the effect of UV radiation on the adhesive within its open time.

Mechanical stress, elevated temperatures, and high relative humidity can be a fatal combination for certain adhesives and sealants if all occur at the same time. As the temperature increases, the bond strength decreases.

Environmental consequences are so severe that it is usually necessary to test preproduction joints, both in the laboratory and in the field, and under conditions as close to the actual service environment as possible. Elevated temperature exposure could cause oxidation or pyrolysis and change the rheological characteristics of the adhesive. Chemical environments cause corrosion at the interface. Inorganic coatings are now widely used for thermal protection and as a protection against high – temperature corrosion. Nevertheless, there are certain polymers that have excellent resistance to high temperatures over short duration (e.g., several minutes or hours). However, prolonged exposure to elevated temperatures may cause several reactions to occur in the adhesive or sealant**.** The most encountered reactions that occur in the material are oxidation and pyrolysis. Some organic adhesives tend to be susceptible to chemicals and solvents, especially at elevated temperatures. Water is the substance that gives the greatest problems in terms of environmental stability for many adhesive joints.

Many polymers (especially epoxies) are hydrophillic and absorb moisture from their operating environments. Absorbed moisture reduces Tg, modulus and (usually) strength but may enhance damage tolerance.

The moisture may be absorbed into the adherends or adhesives prior to bonding process.

In elevated temperature cure joints, pre-bond absorbed moisture may affect the performance of the formed joint by:

- interfering with surface wetting and consequential development of interfacial bonds at the adherend/adhesive interface.
- interfering with the cure reaction of the adhesive.
- causing excessive voiding in the adhesive.

1.7 Surface treatments

The cleaning and de-greasing of surfaces is a very important stage in the bonding process, but even so it is not a sufficient condition to obtain bond durability. The substrates must be dry also. A surface, whatever its chemical nature, corresponds a discontinuity of matter. The materials to be bonded must be clean and free of dust or grease. To obtain a maximum mechanical strength, most substrate needs a surface treatment before to the application of adhesive. There are a variety of surface treatment (pre- and post-treatment) as corona discharge, sandblasting treatment, oxygen plasma, acid etching, laser treatment, pickling, low-pressure plasma, SACO process.

General presentation of the various surface treatments

There are three different types of surface treatment: mechanical, chemical and by a combination of these two treatments when the performance must be optimal.

1. Mechanical treatment

Adhesive sanding - this method consists of projecting abrasive particles on the surface treated. The sanding is used to essentially metal surfaces. The abrasive powder is better than sand (SiO_2) because it is less hard and its edges are less “cutting”.

Ultrasonic cleaning - it is a treatment that is, both, mechanical and chemical.

The principle of this method is that the waves are issued by the vibration of quartz plates in the ultrasonic field (frequency between 20000 Hz and some Mega – Hz).

With regard to ultrasonic cleaning, the piece should be immersed in liquid condition itself in a special tank, known at “Ultrasonic tank**“.** The spread of the sound wave is reflected by the vibrating molecules of the liquid. Each element of the liquid is subjected to pressure-depression around an equilibrium position. From a certain point the pressure generated is less than the saturation vapor pressure of liquid, and there is formation of vapor bubbles, producing the phenomenon called *cavitation* [10].

2. Chemical treatment

This cleaning may be adequate in the case of non-structural collages, but it must be the first step of treatment in the case of a structural adhesive.
The different types of solvents:

- Aromatic hydrocarbons
- Aliphatic hydrocarbons
- Aliphatic halogenated hydrocarbons

Heat cleaning is the most performante; it can be done by immersion or vapor phase.

Chapter 2

2.1 Fracture mechanics – History and introductory notions

From investigating fallen structures, engineers found that most failure began with cracks. These cracks may be caused by material defects (dislocation, impurities), discontinuities in assembly and/or design (sharp corners, grooves, and voids), harsh environments (thermal stress, corrosion) and damages in service (impact, fatigue, unexpected loads). Most microscopic cracks are arrested inside the material but it takes one run-away crack to destroy the whole structure.

..Every structure contains small flaws whose size and distribution are dependent upon the material and its processing. These may vary from nonmetallic inclusions and micro voids to weld defects, grinding cracks, quench cracks, surface laps, etc. [11].

Fracture mechanics was introduced to analyze the relationship among stresses, cracks, and fracture toughness. The objective of a fracture mechanics analysis is to determine if these small flaws will grow into large enough cracks to cause the component to fail catastrophically. Fracture mechanics is the field of solid mechanics that deals with the behaviour of cracked bodies subjected to stresses and strains.

The first milestone was set by Griffith in his famous 1920 paper that quantitatively relates the flaw size to the fracture stresses [12]. However, Griffith's approach is too primitive for engineering applications and is only good for brittle materials. For ductile materials, the milestone did not come about until Irwin developed the concept of *strain energy release rate*, G, in 1950s [12]. G is defined as the rate of change in potential energy near the crack area for a linear elastic material.

$$G = -\frac{d\pi}{dA} \tag{1}$$

In 1968, Rice modeled the plastic deformation as nonlinear elastic behavior and extended the method of energy release rate to nonlinear materials [12]. He showed that the energy release rate can be expressed as a path-independent line integral, called the *J integral*. Recent trends of fracture research include dynamic and time-dependent fracture on nonlinear materials, fracture mechanics of microstructures, and models related to local, global, and geometry-dependent fractures.

Fracture may occur in three different loading modes: mode I (opening), mode II (forward shear), and mode III (anti-plane or out-of-plane shear or tearing), as shown in Figure 5. Just as strength-based failure criteria become more involved for multiaxial stress fields, the fracture criterion for mixed mode loading may include contributions from each mode in some appropriate manner.

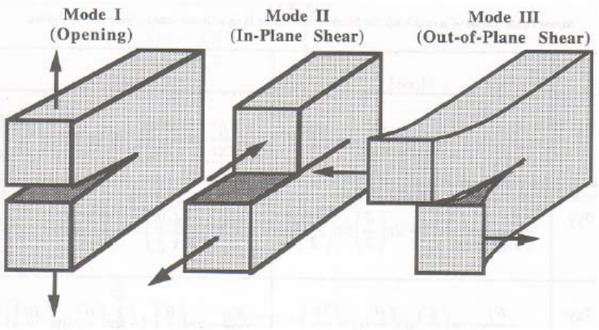


Figure 5 - Fracture modes [13].

Stress Intensity, K, is a parameter that relates the magnitude of the applied load or stress, σ and geometry, represented by the crack length a. In practice, engineers are interested in the maximum stress near the crack tip and whether it exceeds the fracture toughness. Thus, the stress intensity factor K is commonly expressed in terms of the applied stresses σ at $r \to 0$ and $\theta = 0$

For example, for a through crack in an infinite plate under uniform tension σ as shown in Figure 6, the stress intensity factor is:

$$K_1 = \sigma\sqrt{\pi a} \quad (2)$$

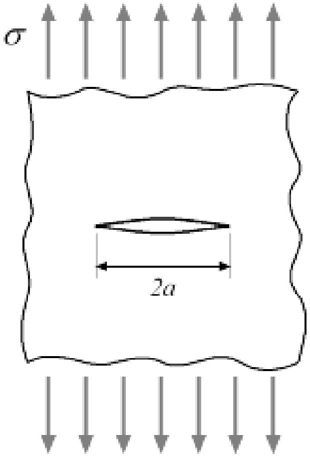


Figure 6 – the stress intensity factor [14].

where a is one half of the width of the through crack. The dimension of K is:

$$Dim[K] = \frac{F}{L^2}\sqrt{L} = F \cdot L^{-\frac{3}{2}} = Stress \cdot \sqrt{Length} \tag{3}$$

Based on the linear theory the stresses at the crack tip are infinity but in reality there is always a plastic zone at the crack tip that limits the stresses to finite values. It is very difficult to model and calculate the actual stresses in the plastic zone and compare them to the maximum allowable stresses of the material to determine whether a crack is going to grow or not. The most experimental method used is to calculate a critical stress intensity factor K_c for each material, called the fracture toughness of the material. One can then determine the crack stability by comparing K and K_c directly.

Stress intensity (K) in any mode situation is directly proportional to the applied load on the material.

The mode I and II stress intensity factors are defined as:

$$K_I = \lim\left\{\sqrt{2\pi r}\ (\sigma_{22})_{\theta=0}\right\} \tag{4}$$

$$K_{II} = \lim\left\{\sqrt{2\pi r}\ (\sigma_{12})_{\theta=0}\right\}$$

where the coefficients K_I and K_{II} are known as the mode I and II stress intensity factors,

The mode III stress intensity factor is introduced to obtain near-tip stresses and displacements:

$$K_{III} = \lim\left\{(2\pi r)^{\frac{1}{2}}\,(\sigma_{32})_{\theta=0}\right\} \tag{5}$$

The energetic criterion is based on the assumption that crack growth will occur when the energy available at the crack tip due to the applied load (*G* – strain energy release rate), overcomes the critical strain energy release rate (*G*c), which is a material property.

The cracks in adhesively bonded systems can be cohesive and/or adhesive. Cracks are considered to be cohesive when they grow entirely within the adhesive layer. When this happens it means that the bond between the adhesive and adherend is performing well.

For a linearly elastic material, and small displacements, the energy release rate G can be written as follows:

$$G = \frac{1}{E}\left({K_I}^2 + {K_{II}}^2\right) + \frac{1}{2\mu}{K_{III}}^2 \qquad (6)$$

where

$$\bar{E} = \frac{E}{1-\vartheta^2}\ for\ plane\ strain\ and\ \bar{E} = E\ for\ plane\ stress$$

For a mode I crack at initiation

$$G_C = \frac{K_{c2}}{\bar{E}} \qquad (7)$$

K_C^2 – fracture toughness.

In the case of adhesive cracking, cracks run along the interface between two materials due to interactions between the stress field in the adhesive layer and spatial variations in fracture properties. The cracks are not generally free to evolve as mode I cracks, as was the case for cohesive cracks, and mixed-mode fracture (combinations of tension and shear) have to be considered. Mode II or shear components are induced, even in what appear to be nominally mode I loadings, due to differences in modul about the interface.

Cracks are not the only sources of high stresses or singularities in bonded systems: there are generally a multitude of so-called bimaterial comers that also create singular stresses as happened in the rocket case failure, shown in Figure 7. They are admittedly of a slightly different nature from those generated by cracks, but nonetheless can often be handled in a similar manner.

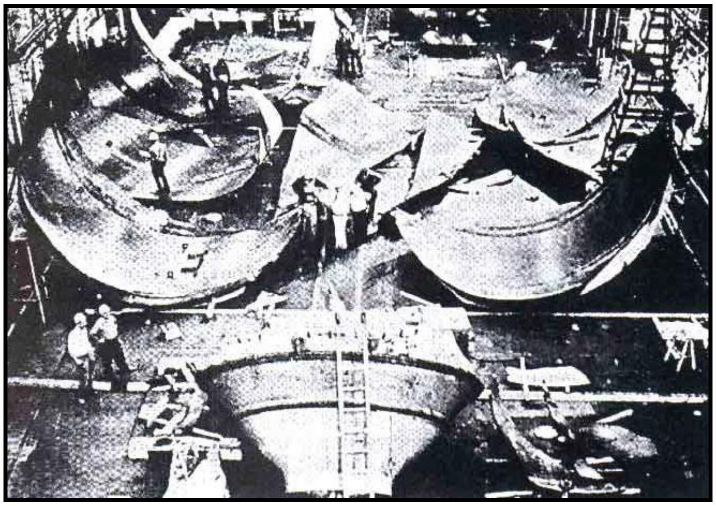


Figure 7 – Rocket Case Failure [15].

2.2 Determination of the toughness of adhesives

A major consideration in the design of adhesively bonded structures is the possibility of crack growth, either within the adhesive or at the adhesive-adherend interface. Crack propagation can be catastrophic when the fracture toughness (*G*c) of the adhesive-adherend system has been exceeded. Debonds or delaminations are probably the most life-limiting defects that occur in layered or laminated structures, and may arise during processing or subsequent service. Common structural features, such as thickness changes, bolt holes or bonded joints, generate through-thickness stress concentrations, which may initiate debonding under static or cyclic loading conditions. The situation is exacerbated at elevated temperatures and in the presence of aggressive environments (eg water vapour or solvents).

Crack initiation and growth usually occurs under mixed-mode conditions, a combination of mode I (crack-opening), mode II (forward-shear) and mode III (scissor-shear) [16, 17]. As a result, fracture toughness tests have been developed to quantify resistance to crack initiation and growth under tensile and shear loading conditions. These test methods are used to assess the influence of adherend surface condition, adhesive, adhesive-adherend interactions, primers, etc., on the fracture strength. Efforts in recent years have been directed towards generating reliable test methods for determining fracture toughness under mixed-mode loading conditions.

Serious doubts have been raised as to the relevance and reliability of employing a fracture mechanics approach for the design and analysis of adhesively bonded structures. It is recognised, however, that fracture mechanics has proved a valuable tool for understanding the fundamental mechanisms of adhesion. Kinloch et al [18] have shown that fracture toughness data obtained from mode I fatigue tests can be used to predict the fatigue life of adhesively bonded joints.

2.2.1 MODE I TESTS

In the compact tension test [19] shaped adherends are bonded together to produce a test specimen with the same geometry as the solid compact tension specimen, Figure 8. Specimens are 25 mm wide with a 25 mm long bond line. The depth of adherend is typically 12 mm at either side of the bond line. The specimen is loaded at one end of the bond producing a peel force. The test is run at a constant loading rate or crosshead speed until the joint is completely failed. The maximum load is recorded.

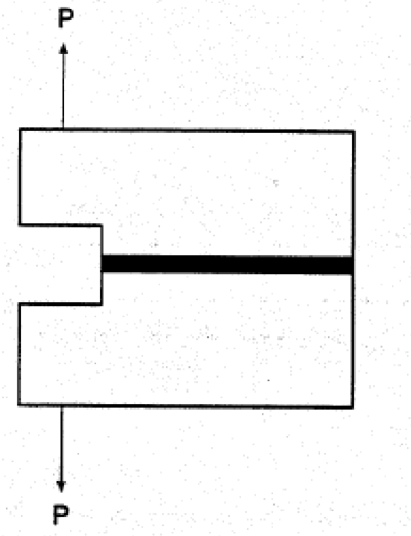


Figure 8 – Compact tension joint specimen.

To calculate the fracture toughness G_{1c} (from load to start of crack), with the ASTM D3433-99 method, the following equation was used:

$$G_{Ic} = \frac{\left[4 \times F^2(\max)\right] \times \left[3 \times a^2 + t_s^{\ 2}\right]}{\left[E \times w^2 \times t_s^{\ 3}\right]} \qquad (8)$$

and the fracture toughness, G_{1a} (from arrest load), as follows:

$$G_{Ia} = \frac{\left[4 \times F^2(\min)\right] \times \left[3 \times a^2 + t_s^{\ 2}\right]}{\left[E \times w^2 \times t_s^{\ 3}\right]} \qquad (9)$$

where:

F (max) = load to start crack, N

F (min) = load at which crack stops growing, N

E = tensile modulus of adherend, MPa

w = specimen width, mm

a = crack length, mm (distance from crack tip to pin hole centers)

t_s = thickness of the adherend, normal to plane of bonding, mm

These equations have been established on the basis of elastic stress analysis, and hold for a sharp-crack condition under severe tensile constraint. It is assumed that the crack-tip plastic region is small compared with the size of the crack (an assumption that may not strictly hold for tough adhesive systems). Thus systems of similar toughness should only be compared when using these equations. Also, the analysis can be used to understand the effect of various environments on a particular type of bonded system.

2.2.2 MODE II TESTS

End Notch Flexure Test

Figure 9 shows the end notch flexure (ENF) specimen [20], which is essentially the doublecantilevered beam specimen loaded in three-point bend. This test characterises mode II (inplane shear) fracture toughness. For small displacements (and negligible transverse shear deformation), strain energy release is calculated:

$$G_{IIc} = \frac{9P^2a^2}{16EB^2h^2} \qquad (10)$$

The ENF test is being developed as a standard method for mode II fracture toughness. Mode II data are important for predicting the behaviour of loading situations where mode I does not dominate.

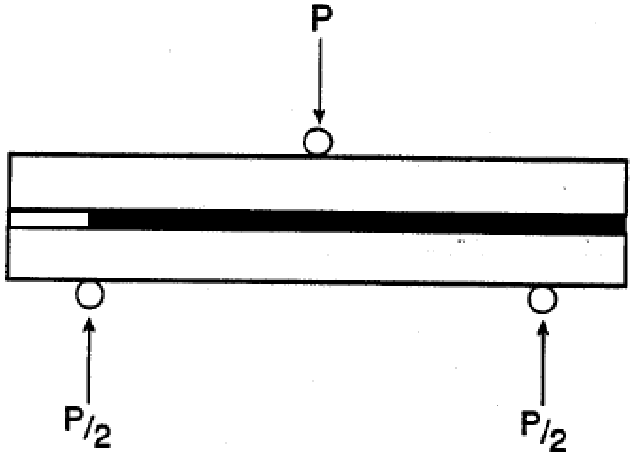


Figure 9 - End Notch Flexure Test.

2.2.3 MIXED-MODE I/II

The problems associated with mode I and mode II loading configurations, such as inelastic material behaviour, large displacement effects, frictional forces in the form of abrasion between upper and lower fracture surfaces are just as pertinent, if not more so, to mixedmode configurations. The work on the following test geometries can be considered still in its infancy, despite the claims of their authors. Further research into measurement techniques for monitoring the onset and progress of delamination is required. This comment also applies to precracking with particular emphasis on quantifying geometrical nonlinearities.

Mixed-Mode Flexure (MMF): This test consists of clamping one end of a DCB specimen and applying load to one of the adherends. The ratios of modes (G_I/G_{II}) is varied by using adherends of different bending stiffenesses (ie altering the thickness ratio of the two adherends).

A schematic of the loading configuration is shown in Figure 10. Test specimen dimensions and fabrication are similar to those used for DCB and ENF specimens. A special fixture is required.

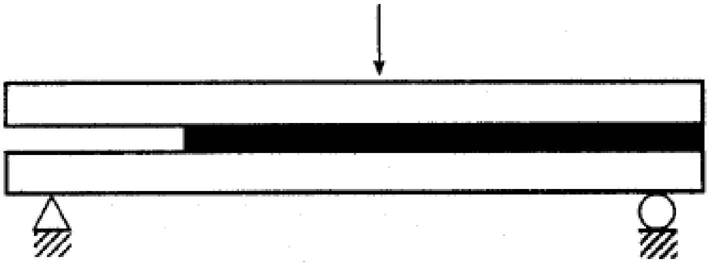


Figure 10 – Mixed mode flexure configuration.

Crack Lap Shear (CLS): This method enables the magnitude of the shear and peel stress components to be varied, by changing the relative thickness of strap and lap adherends. Load is applied axially. Failure is mode II dominated with (GI/GII) ratios ranging from 20 to 35% [20]. The specimen can be loaded in either tension or flexure (fourpoint bend). Mode I component increases when the CLS specimen is subjected to pure bending, also the energy release rate is independent of crack length.

2.3 Double Cantilever Beam test (DCB)

To measure the fracture energy of an adhesive bond experimentally precracked specimens are typically loaded under prescribed conditions allowing the debond to propagate. The double-cantilever beam (DCB) specimen as shown in Figure 11 is a standard specimen (ASTM D3433 -

99) where a_o is considered a region of initial length pre- crack, used to obtain the mode I fracture energy of the adhesive bonds, which is a measure of the fracture toughness of adhesive in the presence of flaws. Within the scope of linear elastic fracture mechanics (LEFM) the DCB specimen is treated as a slender beam.

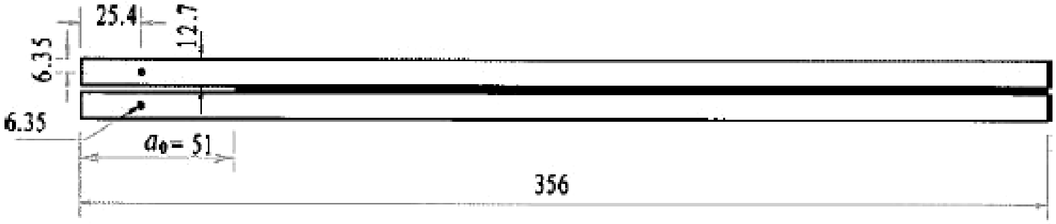


Figure 11 - Geometry of DCB specimen in ASTM D3433-99(dimensions in mm) [21].

Researchers provided a large amount of theoretical analyses on the DCB specimen. An advanced solution for the DCB specimen was developed by Kanninen, applying a Timoshenko beam on Winkler-type elastic foundation [19]. A schematic representation of the DCB test is shown in the Figure12. During the test there is a scroll over P and δ values for the corresponding length of crack a [21].

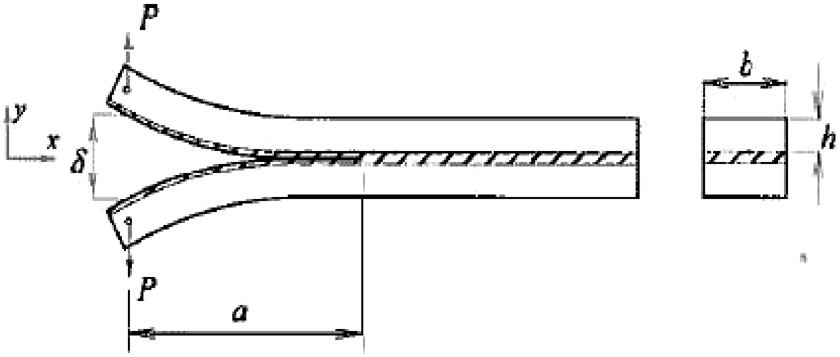


Figure 12 - Schematic representation of the DCB test.

Usually a crack is initiated first by inserting a wedge. The specimen is then loaded by pulling apart the two beams at a certain rate, this increasing load resulting in increased deflection of two beams. At a certain critical load the crack begins to propagate resulting in a slight drop in the load. At this point, the beams are stopped from moving apart, thus keeping the deflection constant. The drop in load (due to increasing crack length) and the crack length are carefully followed. Following the equilibration of the crack, the specimen is consecutively unloaded and then loaded. Ideally, the compliance of the fixture should remain the same during these two cycles if there is no further propagation of the crack.

This overall procedure is repeated several times leading to total cleavage of the specimen. The data finally collected at various times consists of load, deflection, crack length and the compliance. This data can then be analyzed using several different approaches.

In the corrected beam theory approach, the fracture energy is given by:

$$G_{IC} = \frac{3P_{c^2}}{2w} m^3 (a + \widehat{a})^2 \tag{11}$$

where:

P_c is the critical load;

w is the adherend and bond width;

m and $\widehat{a}$ are the slope and debond length correction.

Advantages and disadvantages of the Double Cantilever Beam test is shown in Table 3 [26]:

Table 3-Avantages and disadvantages of the DCB test [26]

Advantages	***Disadvantages***
Yields mode I fracture toughness	Limited to rigid adherents
Compatible with metals and PMCs	Loading tabs and test fixture required
Straight forward/economic specimen testing and fabrication	Moderate to large uncertainties in measurements
Straight forward/economic specimen data reduction	Analysis required to account for end (loading) block tilting, large beam deflection
ASTM D 3433/draft BSI under review	Crack extension measurements difficult
Suitable for cyclic/environmental testin testing	Non linear load-displacement

Tapered Double-Cantilever Beam (TDCB): The purpose of this test geometry is to make the measurement of fracture toughness (G_I) independent of the crack length (ie constant compliance). The specimen design is well suited to tests where the crack length (a) is difficult to measure, especially environmental testing. The critical strain-energy release rate or fracture toughness (G_{Ic}) is calculated as follows:

$$G_{IC} = \frac{4P^2}{Eb^2} \qquad (12)$$

where (P) is applied load, (E) is the Young's modulus of adherend, (b) is the specimen width, (a) is the crack length and (h) is the adherend thickness. A schematic of the TDCB specimen is shown in Figure 13.

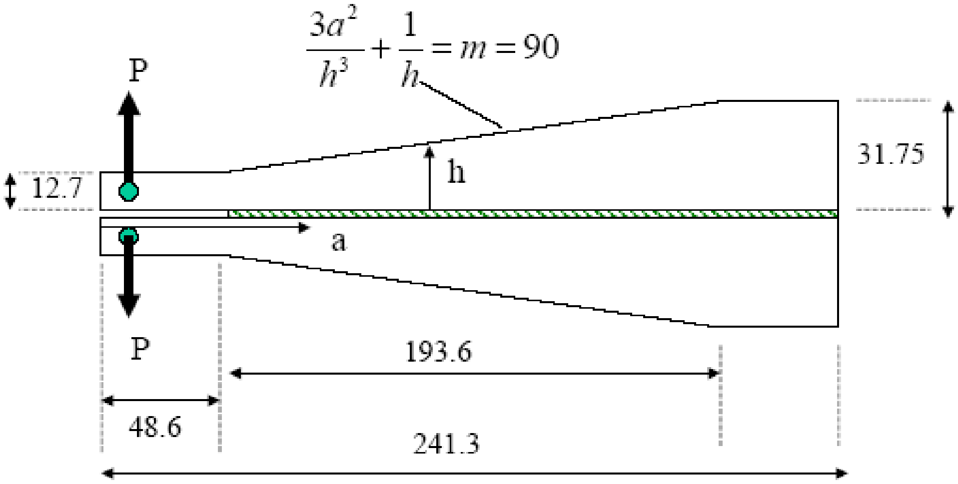


Figure 13 - Tapered double cantilevered beam (TDCB) test specimen [22].

The taper height is chosen such that m is constant with crack length from the relationship:

$$m = \frac{3a^2}{h^3} + \frac{1}{h} \tag{13}$$

The TDCB test specimen has been used to determine the rate of crack growth under various cyclic loading and environmental conditions [22, 23]. Although TDCB adherends and specimen fabrication are more expensive than the DCB specimens, the testing is much more routine and, provided that the adherend materials have high yield stress, the adherends should be reusable. TDCB tests have been used also to assess environmental durability. As with all fracture tests, information on the strength of the interface can only be obtained if the crack propagates along a surface.

Table 4-Avantages and disadvantages of the TDCB test [24]

Advantages	***Disadvantages***
Yields mode I fracture toughness	*Limited to rigid adherents*
Compatible with metals	*Large specimens required*
Adaptable to thin adherends	*Not suitable for generating design data*
Constant compliance	*Special test fixture required*
Straightforward/economic *- specimen fabrication* *- testing* *- data reduction*	*Special glueing fixture required*
Suitable for cyclic/environmental testing	*Moderate to large uncertainties in measurements*

Specimens are typically 241 mm long and 25 mm wide. Adherend thickness has a nominal maximum thickness of 32 mm [25]. Variations of TCDB test are being developed to test thin metal sheet or composite laminates.

Advantages and disadvantages of Tapered double cantilevered beam test are shown in Table 4 [24].

The asymmetric Double Cantilever Beam (ADCB) test

The Asymmetric Double Cantilever Beam (ADCB) test is a generalization of the standard DCB test, where the two arms of the specimen are made of different materials and/or have different thicknesses. The test is suitable for assessing the mixed-mode delamination toughness of laminated composites, as well as the mixed-mode fracture toughness of adhesively bonded joints as shown in the figures below:

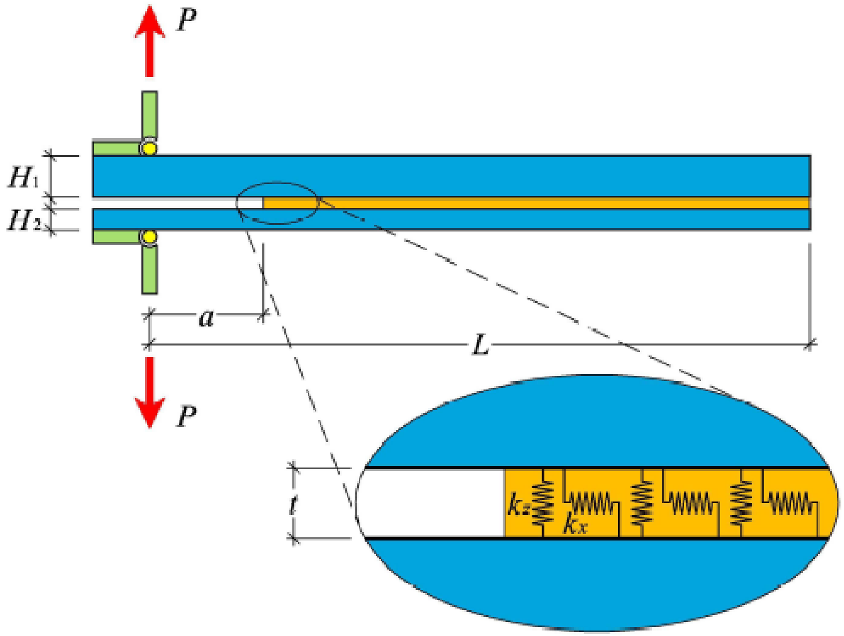


Figure 14 – Asymmetric Double Cantilever Beam (ADCB) [26].

The ADCB test was originally developed by Hugh Brown and has been widely used to measure the fracture energy of polymer-polymer interfaces. The advantage of the ADCB test is that with suitable design of the modulus and thickness of the two adherends, the crack can be forced to propagate close to the interface so that the effects of the surface pretreatment and sol-gel processing conditions can be directly evaluated.

Chapter 3

3. Experimental programme

3.1 Introduction

This study is about the mechanical characterization of adhesives for high temperature, in aerospace applications. The experimental part of this project was based on making the test specimens, testing them, and mechanical interpretation of the results of three test procedures.

A RTV106, a high temperature acetoxy adhesive sealant supplied by GE Bayer Silicones was used. One of the applications of RTV adhesives is to bond the ceramic tiles to the internal aluminium fuselage of the space shuttle.

The following tests were carried out: Thick adherend shear test (TAST), Single lap joint test (SLJ) and Double cantilever beam test (DCB). The DCB test was initially the only test to perform but it was decided to complement the fracture toughness with the shear properties of the adhesive.

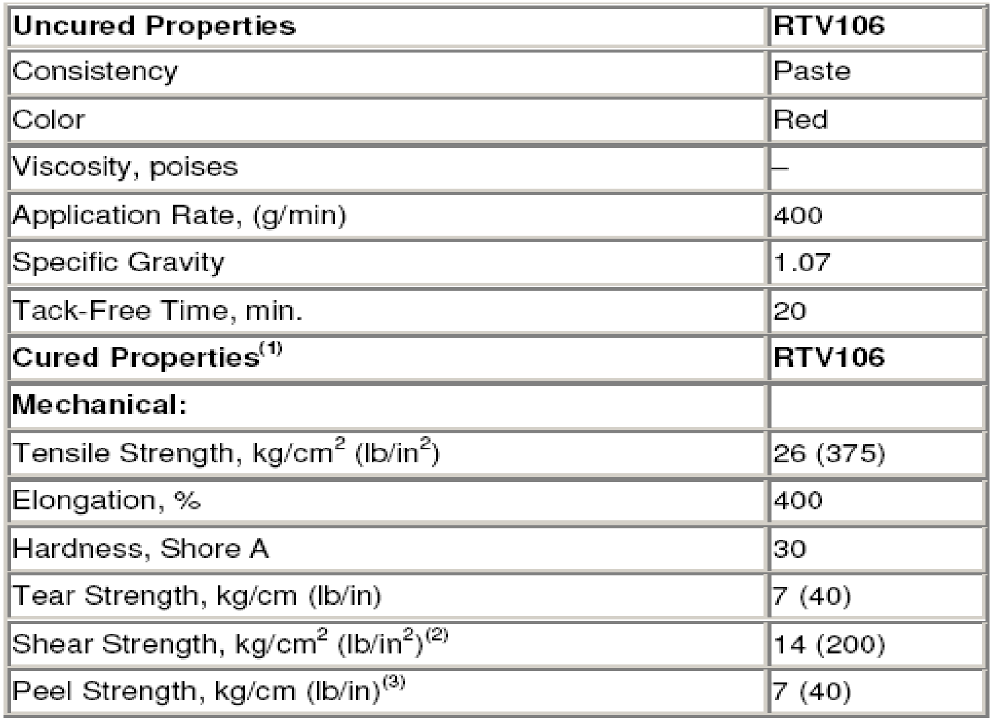
3.2 Adhesive sealant description

The product RTV106 one-component, ready-to-use adhesive sealant is extremely versatile. It cures to a tough, durable, resilient silicone rubber on exposure to atmospheric moisture at room temperature. Acetic acid vapors are released from the sealant surface as a by-product of cure. RTV106 sealant is paste-consistency sealant and is standard strength high-temperature sealant.The key performance properties of this adhesive are [28]:

- ✓ One-component products
- ✓ Capability to cure at room temperature and ambient humidity
- ✓ Self adhesion properties
- ✓ Low temperature flexibility
- ✓ High temperature performance
- ✓ Excellent weatherability and ozone and chemical resistance
- ✓ Excellent electrical insulation properties

RTV 106 sealant is used in aerospace industry but also in other applications which require a high temperature as sealing heating elements, gasketing, electrical insulation, and other critical bonding and sealing applications. It can be applied to vertical or overhead surfaces. Typical product data are presented in Table 5:

Table 5 Typical product data of RTV 106 [28]



Uncured Properties	**RTV106**
Consistency	Paste
Color	Red
Viscosity, poises	–
Application Rate, (g/min)	400
Specific Gravity	1.07
Tack-Free Time, min.	20
Cured Properties$^{(1)}$	**RTV106**
Mechanical:	
Tensile Strength, kg/cm^2 (lb/in^2)	26 (375)
Elongation, %	400
Hardness, Shore A	30
Tear Strength, kg/cm (lb/in)	7 (40)
Shear Strength, kg/cm^2 (lb/in^2)$^{(2)}$	14 (200)
Peel Strength, kg/cm (lb/in)$^{(3)}$	7 (40)

(1) Cure time 3 days/25 ºC (77F)/50% relative humidity.
(2) At 100% cohesive failure.
(3) At 100% cohesive failure using 1 in. x 8 in. stainless steel screen at 180° pull angle.

In addition to the effects of temperature and relative humidity, development of maximum bond strength will depend on joint configuration, degree of confinement, sealant thickness and substrate porosity.

Normally, sufficient bond strength will develop in 12 to 24 hours to permit handling of parts. Minimum stress should be applied to the bonded joint until full adhesive strength is developed. Eventually the adhesive strength of the bond will exceed the cohesive strength of the silicone rubber sealant itself. Always allow maximum cure time available for best results [28].

3.3 Joint manufacture

3.3.1 Thick Adherend Shear Test

The transfer of load between adherends is most effectively achieved by shear stress transfer. The ASTM and ISO standards will quickly reveal a multitude of shear test geometries, each with the objective of producing a state of pure uniform shear in the adhesive layer. In order to access the shear stress–strain behaviour of polymeric materials, there are several experimental methods:

1. V-notched beam shear method (Iosipescu)
2. Notched plate shear method (Arcan)
3. Torsion of bulk material
4. Butt torsion (napkin ring or solid specimen)

5. Plate -twist
6. Thick- adherend shear test method (TAST)

In this experimental work the thick-adherend shear test was used to enable the measurement of the shear properties of the adhesive according to the standard ISO 11003-2:1993 [29]. The TAST is preferred for determining design parameters as the thick, rigid adherends reduce (but not eliminate) the peel stresses. The typical joint geometry is shown in Figure 14. The state of stress is predominantly shear but there are peel stresses at the end of the overlap. Failure occurs at the end of the bond line and is thus sensitive to the geometry of this region. The locus of failure tends to be close to the adherend and, thus, interfacial failure is a possibility. Cracks have been observed to run along the interface. Higher joint extensions can be realised if the corners of the adherends and the spew fillet are profiled to remove stress concentrations. Stresses at the overlap ends are complex and difficult to calculate analytically, although FE approaches have been used [30].

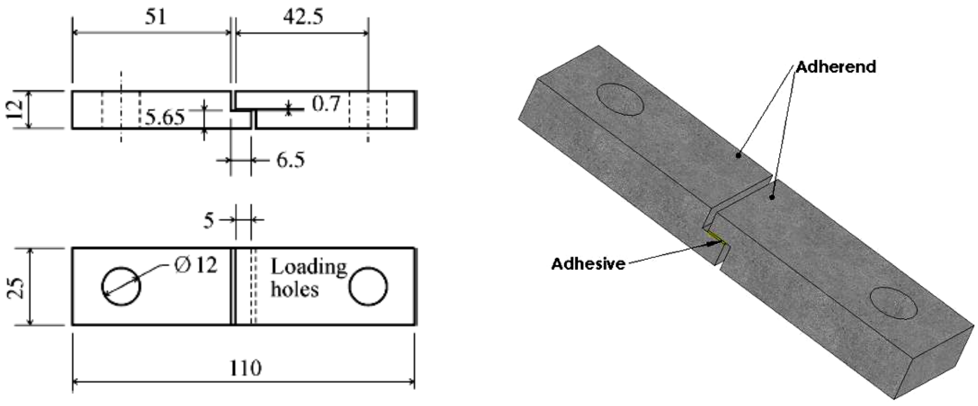


Figure 14 - Test standard TAST specimen (dimensions in mm) [31].

A good specimen is fundamental to achieve cohesive failure and obtain the best test data. To meet this objective the following steps must be followed:

a.) Mould Preparation;
b.) Substrate preparation;
c.) Adhesive mixture (when needed) and deposition;
d.) Hydraulic press;
e.) Curing of the adhesive;
f.) Removal and cleaning;
g.) Preparation for testing.

a.) Mould preparation

The mould used to prepare de bonded joints is illustrated in Figure 15. With this mould we can make up to six specimens in one stage of work.

Figure 15- Mould used to produce TAST specimens.

The first step before starting used the mould in order to produce de bonded joints is to clean it from any burs and remains portions of adhesive. Cleaning is made using an aluminum spatula and then pass acetone using a clean cloth.

b.) Substrates preparation

Steel substrates of dimensions 110 x 25 x 12 mm^3 (Figure 16) were used.

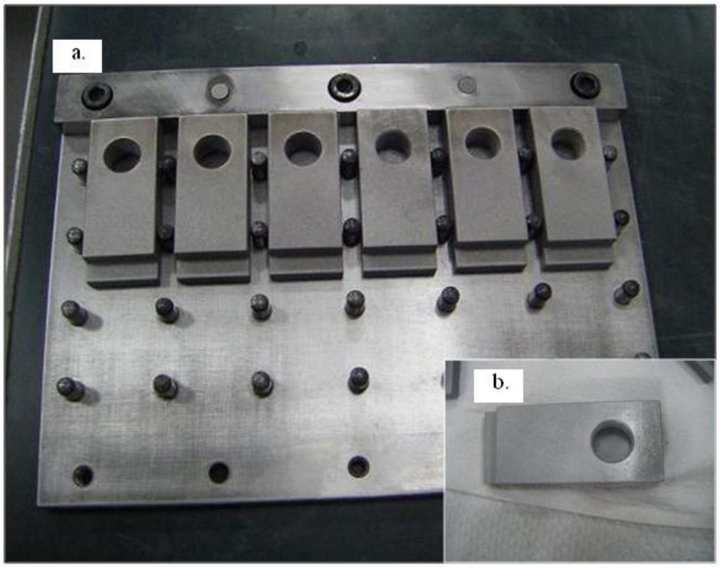


Figure 16 – a.) Six substrates placed in mould; b.) Substrate detail.

The substrates must be treated mechanically prior to the application of the adhesive. Sand blasting is a common treatment, allowing to obtain a good surface for adhesion. This method consists of projecting abrasive particles on the surface treated.

The substrates are placed inside the sandblast machine and the surface was sandblasted with a pressure sufficient to remove all impurities, but the surface should be sandblasted in a manner that the final finishing looks like sand, with high rugosity (Figure 17).

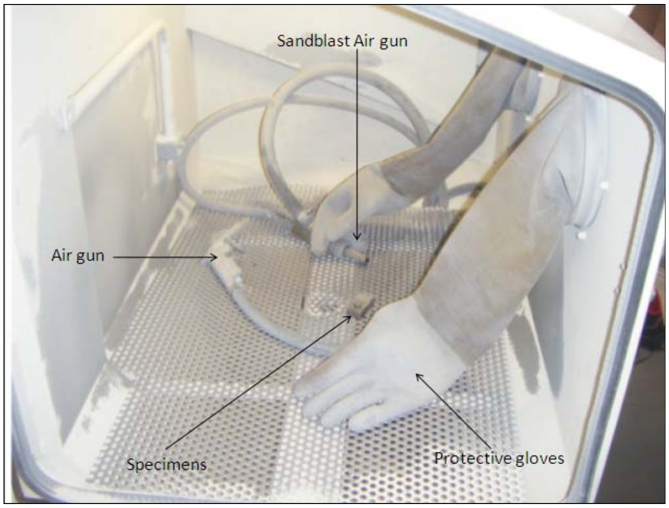


Figure 17 - Specimens placement inside the sandblasting machine in Instituto de Engenharia Mecanica de Gestao Industrial INEGI.

The sandblasting machine was conceived and realized by NORBLAST di Norelli Bologna 2001, and has the following technical characteristics : model S9, minimum pressure 2 bar, maximum pressure 8 bar, line voltage 230V, frequency 50Hz, current 0.6 A (Figure 18).

Figure 18 - Sanblasting machine.

Figure 19 shows the operator with his hands in the sandblaster protective gloves, looking through the screen and pressing the foot actuator for the jet airflow.

Figure 19 - Working with the sandblaster.

c.) Adhesive mixture (when needed) and deposition

An important step before to put an adhesive on the mould is the release agent. The release agent was applied three times on each surfaces of the mould that was in contact with the adhesive joint. The application of release agent guarantees there is not adherence of the adhesive to the mould. The product that was used is Frekote ® 770-NC (Figure 20).

Figure 20 - Application of release agent in a ventilated place.

Figure 21 – RTV 106 Adhesive.

Figure 22 shows how the mould looks like after adhesive was applied using a special gun.

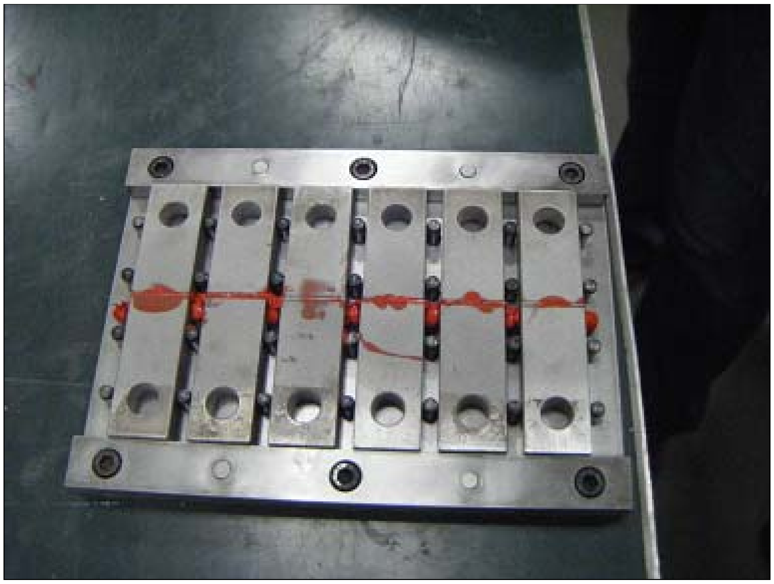


Figure 22 – TAST specimens in the mould.

d.) Hydraulic press

The mould was put in a hydraulic heated press (Figure 23) (Intoco).

Figure 23 – Hydraulic press Intoco.

The mould must be carefully placed in the press not to have a displacement of the specimens.

e.) Curing of the adhesive

The temperature was set in accordance to specifications provided by the manufacturer. In the following table are given some specifications for RTV 106.

Table 6 - Temperature/humidity and curing time

Adhesive	*Temperature* [ºC] and *Humidity*	*Curing time*	
		in the press	outside the press
RTV 106	25[ºC] Relative 50%	24 Hours	3 days

f.) *Removal and cleaning*

After the curing time in the press, the mould was removed and the specimens were taken out the mold.

Cleaning and removing the excess of adhesive that remains in the specimen sides must be done only after the curing time outside the press is over. A knife or a piece of aluminum can be used to remove the excess of adhesive.

g.) *Preparation for testing*

Once the specimen is free from the adhesive in excess, the bond line must be clearly visible and have a constant thickness.

3.3.2 Single lap joint test (SLJ)

Single lap joint tests, [32-38], are relatively straightforward from the specimen preparation and testing perspective. Single lap joints create bending loads in the adherends and tensile stresses in the adhesive in addition to the shear stress with the result that the joint becomes very inefficient. These tests have recognised limitations for the accurate determination of joint design parameters. These stresses are highly non-linear, increasing rapidly near the ends. The stress state in this region is highly complex. The directions and magnitudes of these stresses depend on numerous factors:

- applied load and deflection (increasing either leads to an increase in stress)
- mechanical response of the adhesive (elastic or elastic-plastic behaviour)
- mechanical stiffness of the adherends (lower stiffness leads to higher peel stresses) [32]
- geometry at end of overlap (shape of adherend corners, joint fillet) [33 -38]

There are some closed-form analytical solutions for the adhesive stress along the overlap [33, 35] but these require idealised geometries and experimental verification has proved difficult to obtain. Finite element (FE) methods can be used to predict stress concentrations for any geometry [32, 33].

The single-lap specimen is easy to prepare and test. A fixture is used to ensure correct overlap and accurate alignment of the adherend. This may include control of the fillet.

The geometry of the lap shear joint specimens used is shown in Figure 24.

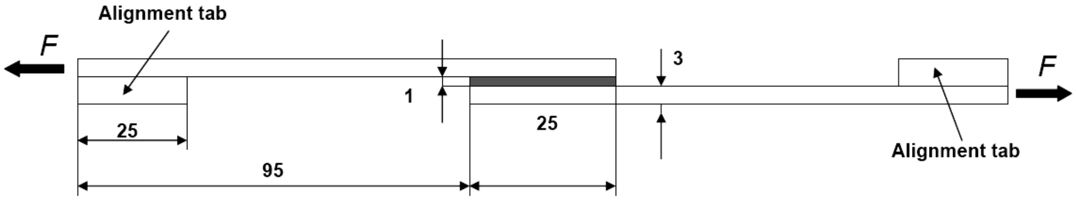


Figure 24 - Lap joint specimen geometry (dimenssion in mm).

Testing can be conducted using standard tension/compression mechanical test equipment. The lap-shear strength (τ) is given by:

$$\tau = P/bL \tag{14}$$

where (P) is the maximum load , (b) is the joint width and (L) is the overlap length.

The main problem with the single-lap shear test is that the average shear strength determined using this method does not correspond to a unique material property of the adhesive, and therefore cannot be used as a design parameter In fact, this quantity is strongly dependent on the joint geometry.

Specimen Manufacture

As in previous test case (TAST), for single lap joint (SLJ) specimen preparation there are seven main stages, too:

a.) Mould Preparation;
b.) Substrates preparation;
c.) Adhesive mixture (when needed) and deposition;
d.) Hydraulic press;
e.) Curing of the adhesive;
f.) Removal and cleaning;
g.) Preparation for testing.

a.) Mould preparation

The mould used for this test is presented in Figure 25. The mould has to be cleaned from any burs and remains portions of adhesive using an aluminium spatula like in Figure 16.

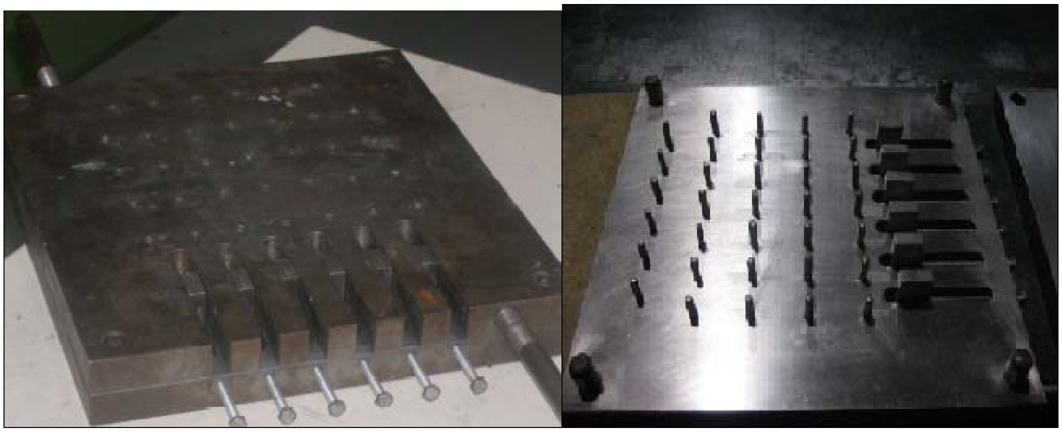


Figure 25 - Mould used for SLJs.

b.) Substrate preparation

High strength steel substrates of dimensions 107.5 x 25 x 2 mm^3 were used in order to avoid plastic deformation of the adherends. The joint surfaces were grit blased and degreased with acetone prior to the application of the adhesive. Sand blasting is a common treatment, allowing to obtain a good surface for adhesion. Substrates are sandblasted (Figure 20) on both sides to avoid deformation. After treatment the specimens look like in Figure 26.

Figure 26 – Sandblasted substrates for SLJs.

c.) Adhesive mixture (when needed) and deposition

At this stage the release agent Frekote 770 - NC is applied as shown in Figure 27.

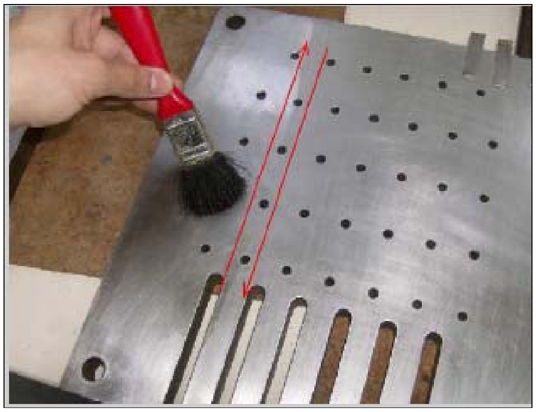


Figure 27 – Release agent application to the SLJs mould.

The next operation is to place the tab ends, adherents and spacers in the mould (Figure 28 - 30). After that, a generous amount of adhesive quantity is applied in the overlap length and in the tab ends bonding area (Figure 31). This can be done using a spatula.

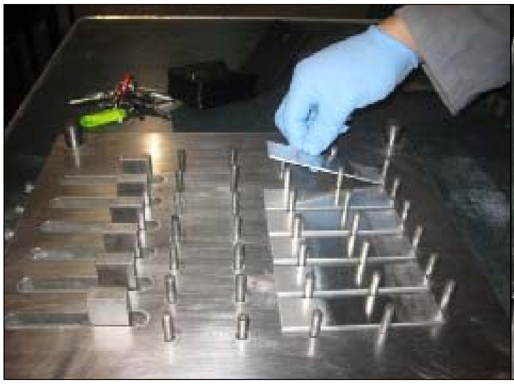


Figure 28 – Adherends placement.

Figure 29 – Spacers placement.

Figure 30 – Tab-ends placement

Figure 31 - Adhesive application

d.) Hydraulic press

The substrates were bonded and then the specimens were cured in a hydraulic press Intoco (Figure 25) following the manufacturers curing conditions.

e.) Curing of the adhesive

The cure followed is shown in Table 6.

f.) Removal and cleaning

After the curing time in the press, the mould was removed and the specimens was taken out the mold. There may be the need for removing the pins of the mold (Figure 32) to facilitate the extraction of the joints.

Figure 32 - Mould pin removal.

The specimens were generally bonded together. It was necessary to separate them with a fine saw. After all the specimens were separated, the adhesive excess was removed. The adhesive was removed with a large woodworking file and when close to the adherends, a smaller woodworking file was used (Figure 33).

Figure 33 - Adhesive removal with a small woodworking file.

g.) Preparation for testing

The joints were wrapped in aluminum foil and put the in a desiccator, avoiding the change of adhesive properties from contact with air humidity.

3.3.3 Double cantilever beam test (DCB)

The double-cantilevered beam (DCB) test [25], Figure 36, is used to measure the initiation and propagation energy of a mode I crack. The critical strain energy release rate or fracture toughness, G_{IC}, depends on crack length and is calculated by:

$$G_{IC} = \frac{4P^2(3a^2 + h^2)}{Eb^2h^3} \qquad (15)$$

Where: P is the applied load in (N), E is the Young's modulus of the adherend (in MPa), b is the specimen width (mm), a, is the crack length (mm) and h is the adherend thickness (mm). Specimen preparation is straightforward. The thick adherend parts can be bonded and tested directly. When the substrates are formed from thin sheet material end tabs need to be attached to allow the joints to be gripped. The dependence of specimen compliance and strain energy release on crack length adds to the complexity and reliability of carrying out and analyzing this test.

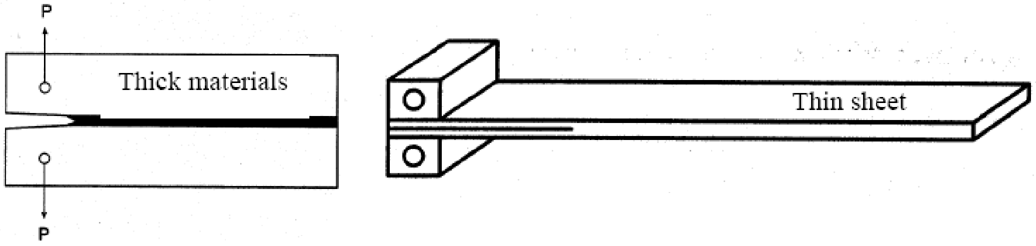


Figure 34 – Double cantilever beam (DCB) test.

Specimen Manufacture

For double cantilever beam (DCB) specimen preparation there are eight main stages:

a.) Mould Preparation;
b.) Substrates preparation;
c.) Spacers and razor blade preparation;
d.) Adhesive mixture (when needed) and deposition;
e.) Hydraulic press;
f.) Curing of the adhesive;
g.) Removal and cleaning;
h.) Preparation for testing.

a.) Mould preparation

The mould used for this test was the same as that used for SLJs (Figure 27).

b.) Substrates preparation;

The steels substrates shown in Figure 35 were used.

Figure 35- Steel substrates used for DCB test.

The beams must be prepared before applying the adhesive. Sand blasting is a common treatment, allowing obtaining a good surface for adhesion.

Figure 36 shows the substrates after the sandblasted surface preparation.

Figure 36 - Sandblasted surface in the substrates.

c.) Spacers and razor blade preparation

The bondline thickness was calibrated with a spacer in the top end and a razor blade was used for the initial crack length tip. This is an important step of the specimens manufacture, because it is crucial to obtain a correct thickness for the adhesive layer.

To obtain 1 mm adhesive layer thickness, the following combination was used:

- Razor blade bottom spacer : 1x 0.4 mm + 1x 0.05 mm
- Razor blade upper spacer :1x 0.4 mm + 1x 0.05 mm
- Razor blade thickness : 0.1mm

This make a bond line thickness of 1 mm with a crack of 0.1 mm in the middle.

Figure 37 - Razor blades used to promote the initiation of the crack.

Razorblades were cut in two symmetric blades. This allows using one razorblade for two crack initiation spacers (Figure 38).

Figure 38 –Measuring and cutting of the razorblade in two symmetric blades.

The first operation is to bond the razorblade to the bottom spacer of 0.05mm. In the second operation the razorblade was bonded to the bottom spacer is bonded to the top spacer resulting the assembly (Figure 39).

Figure 39- Operation of razor blade spacer bonding.

d.) Adhesive mixture (when needed) and deposition

The release agent Frekote 770- NC was applied.

The next operation was cleaning with a paper tissue the substrate and placing the bottom substrate on the mould (Figure 40). After that the adhesive was applied (Figure 41).

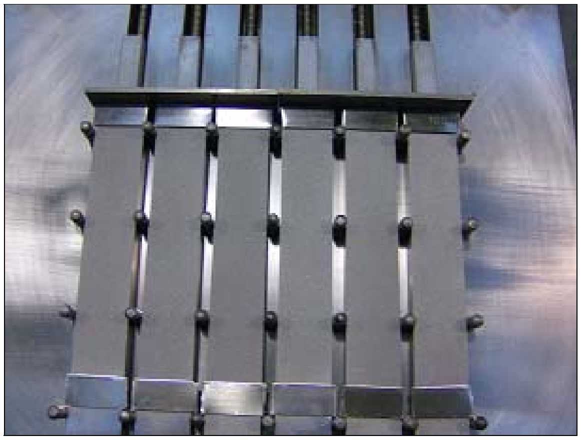


Figure 40 – Bottom substrate in the mould

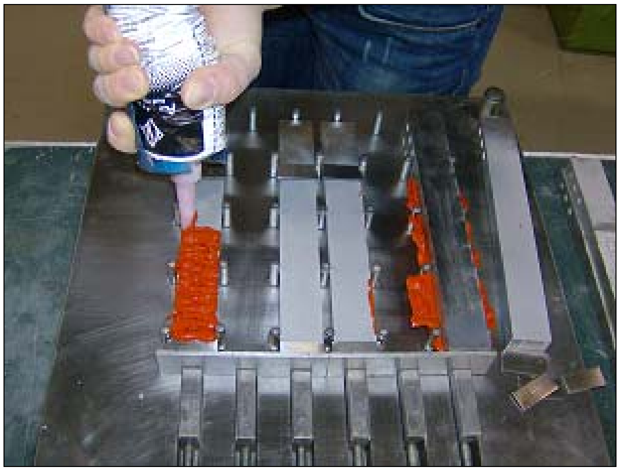


Figure 41- RTV 106 application

The next operation was to place and press the top substrate on the mould (Figure 42).

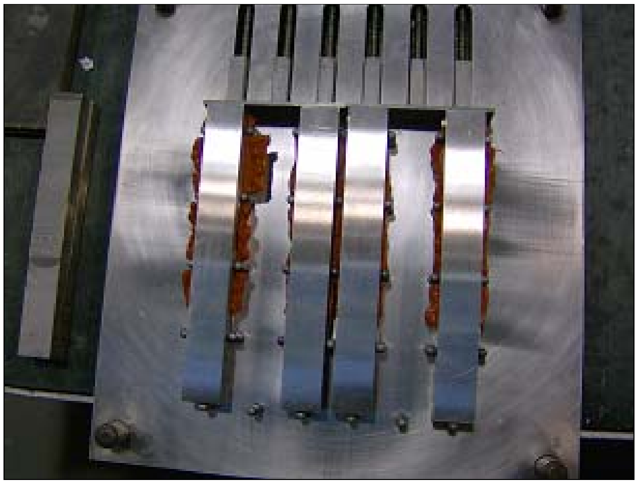


Figure 42 –Complete DCB joints in the mould

e.) Hydraulic press

The mould with the specimens was put into the hydraulic heated press (Intoco).

f.) Curing of the adhesive

g.) Removal and cleaning

After the curing time in the press, the mould was removed and the specimens were taken out the mold. There may be the need for removing the pins of the mold. A knife or a piece of aluminum can be used to remove the excess of adhesive.

h.) Preparation for testing

Once the specimen was free from the adhesive in excess, the bonline was clearly visible with a constant thickness.

3.4 Test method

3.4.1. TAST

The TAST was performed at room temperature on a MTS servo-hydraulic machine MTS, model 312.31, at a constant crosshead rate of 1 mm/min. For load measurements 10% of the capacity of the load cell (25 kN) was used. The displacement was measured with a 25 mm length MTS extensometer. As the extensometer is mounted in the metallic substrate, the extensometer measures not only the displacement of the adhesive, but also the displacement of the adherend. However, da Silva *et al.* [39] showed that the steel deformation can be neglected in the case of flexible adhesives, so that the adhesives displacement can be measured by the MTS extensometer method. Five joints were tested.

Figure 43 – TAST specimen set-up.

3.4.2 Single lap joint tests

Testing was conducted at room temperature at a constant displacement rate of 1 mm/min using the MTS 312.31 servo-hydraulic machine. Loads and displacements to failure were recorded. Three joints were tested to failure. For each joint tested, load-displacement curves were produced.

Figure 44 – SLJS specimen set-up.

3.4.3 DCB

Tests were conducted at room temperature at a constant displacement rate of 1 mm/min using the MTS 312.31 servo-hydraulic machine (Figure 45). Loads and displacements to failure were recorded. Three joints were tested. The experimental values of the crack length were obtained by direct measuring, with the help of a crack length measurement system. The crack length measurement system has a 0.10 precision.

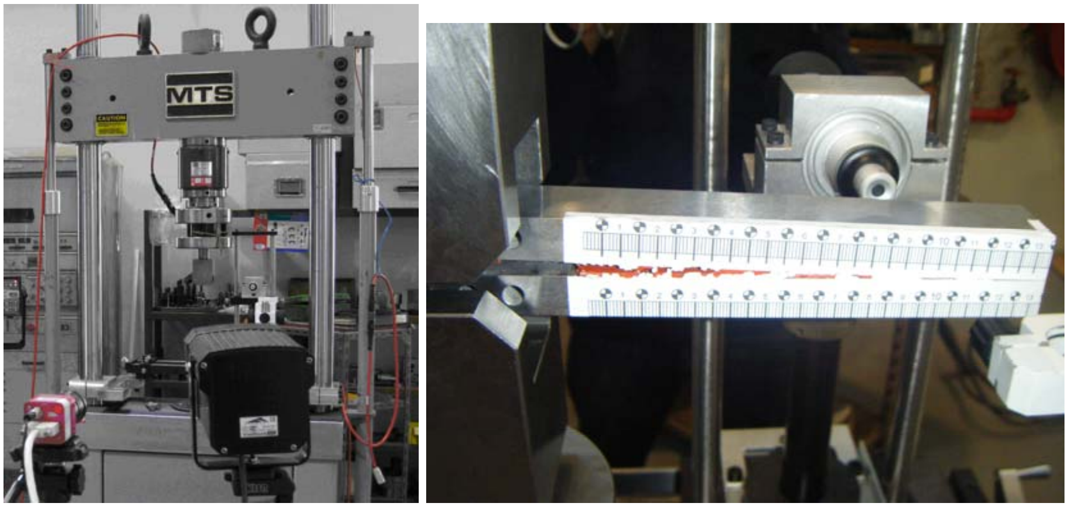


Figure 45 – DCB test set-up.

Chapter 4

4. Results and discussion

4.1. TAST

Typical shear stress-strain curves for the RTV106 adhesive tested at room temperature are shown in Figure 46. From the shear stress-strain curve, the shear modulus and shear strength were calculated. In general elastomeric materials exhibit non–linear stress–strain behaviour and the definition of the modulus is very difficult. However, at small shear strains they obey Hooke's law and the modulus can be found. The values for shear modulus were calculated from the tangent to the shear stress-strain curve at the origin (a polynomial approximation of the curve was made).

Shear strength and strain data for are presented in Table 7.

Table 7. Shear strength and strain data tested at RT.

SHEAR STRENGTH MPA	SHEAR STRAIN %
1.97 ± 0.02	415 ± 30

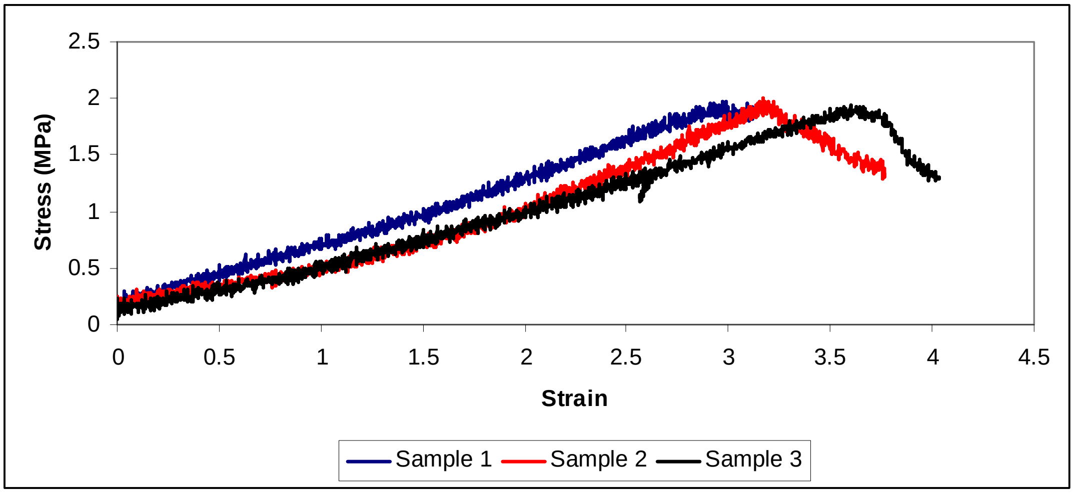


Figure 46 - Typical TAST shear stress-strain curves of RTV 106 adhesive.

Typical failure modes of adhesives in TAST specimens are presented in Figure 47. The failure was a mixed mode (cohesive/adhesive) failure.

Figure 47 - Failure mode in TAST specimens.

4.2. SLJ tests

The average lap-shear strength (τ_{av}) is given by:

$$\tau_{av} = P/bL \tag{14}$$

Where P is the maximum load, b is the joint width and L is the joint overlap length.

Representative load-displacement curves SLJs are presented in Figure 48**.** A summary of maximum loads and average lap shear strengths SLJs tested at RT is presented in Table 8. It can be seen that the adhesive strength from SLJs is approximately the same with the strength from TAST. This is because the stress distribution is uniform in SLJs due to very low stiffness of the adhesive.

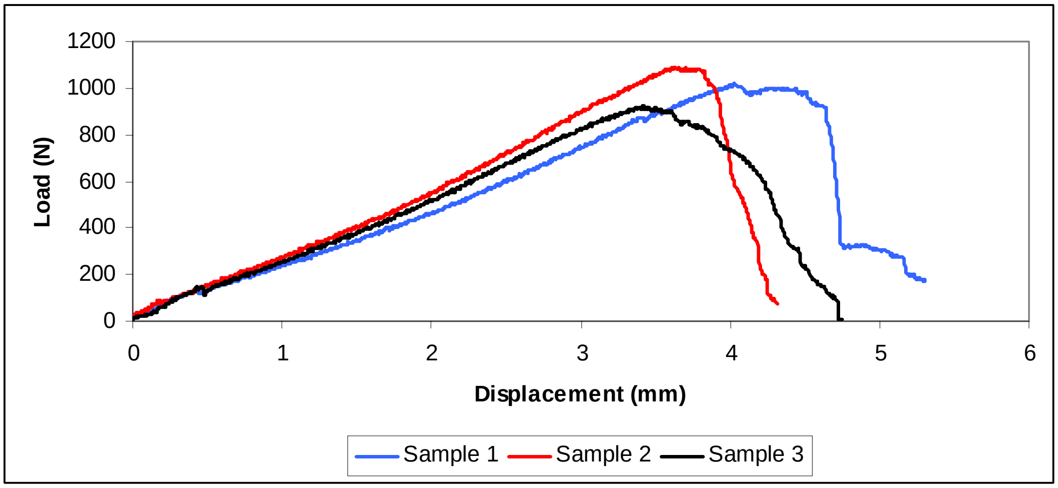


Figure 48 - Typical load- displacement curves of SLJs RTV 106 adhesive.

Table 8. Maximum load and average lap shear strength for RTV106 SLJs tested at RT.

TEMPERATURE ºC	MAXIMUM LOAD (N)	AVERAGE LAP SHEAR STRENGTH (MPA)
RT	1017.66 ± 88.79	1.64 ± 0.13

After the tests, the failure modes of the specimens were evaluated visually. The failure mode was mainly cohesive, with some spots of adhesive failure.

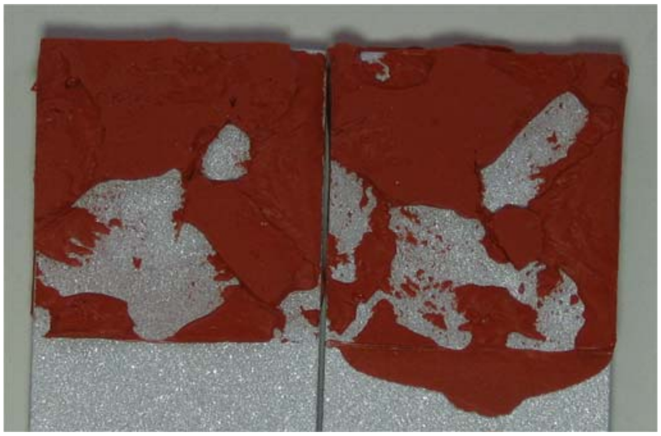


Figure 49 - Failure mode in SLJs.

4.3. DCB tests

To calculate the fracture toughness G_{1c} (from load to start of crack), with the ASTM D3433-99 method, the following equation was used:

$$G_{Ic} = \frac{\left[4 \times F^2(\max)\right] \times \left[3 \times a^2 + {t_s}^2\right]}{\left[E \times w^2 \times {t_s}^3\right]} \qquad (8)$$

and the fracture toughness, G_{1a} (from arrest load), as follows:

$$G_{Ia} = \frac{\left[4 \times F^2(\min)\right] \times \left[3 \times a^2 + t_s{}^2\right]}{\left[E \times w^2 \times t_s{}^3\right]} \qquad (9)$$

where:

F (max) = load to start crack, N

F (min) = load at which crack stops growing, N

E = tensile modulus of adherend, MPa

w = specimen width, mm

a = crack length, mm (distance from crack tip to pin hole centers)

t_s = thickness of the adherend, normal to plane of bonding, mm

The values of the toughness for RTV 106 adhesive can be seen in the Table 9. It can be seen that although the silicone adhesive had low strength, it exhibited high DCB fracture toughness because of a large plastic deformation zone in the fracture process. For example, a strong and brittle epoxy adhesive AV138 has a critical strain energy release rate in mode I G_{Ic} (N/m) of 345.9±47.8 [40].

Table 9. The values of the toughness for RTV106 SLJs tested at RT.

G_{1C} [J/M^2]	G_{1A} [J/M^2]
1151.757 ± 93.78	832.826 ± 274.08

A typical DCB load-displacement curve is presented in Figure 50**.**

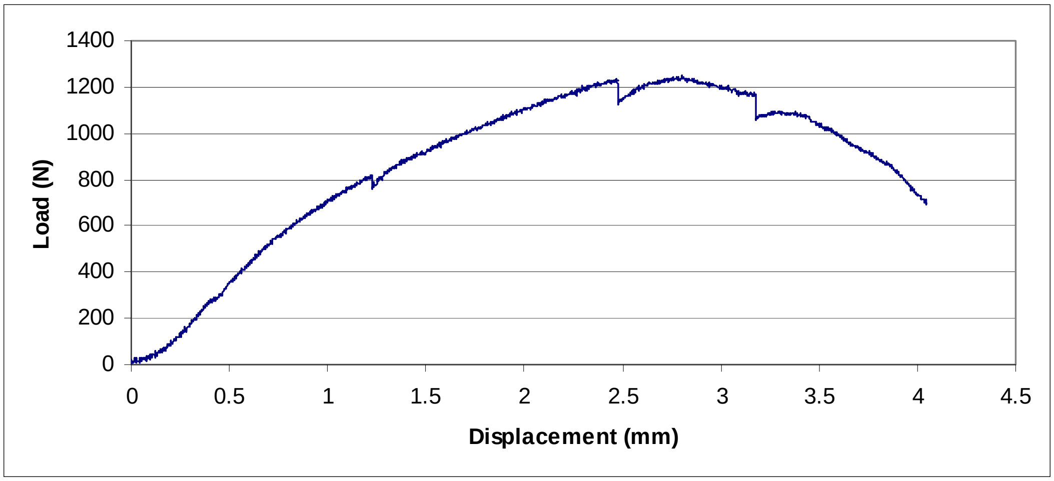


Figure 50 - Typical DCB load-displacement curve

After the tests, the failure modes of the specimens were evaluated visually. The failure mode was mainly cohesive, as can be seen in Figure 51.

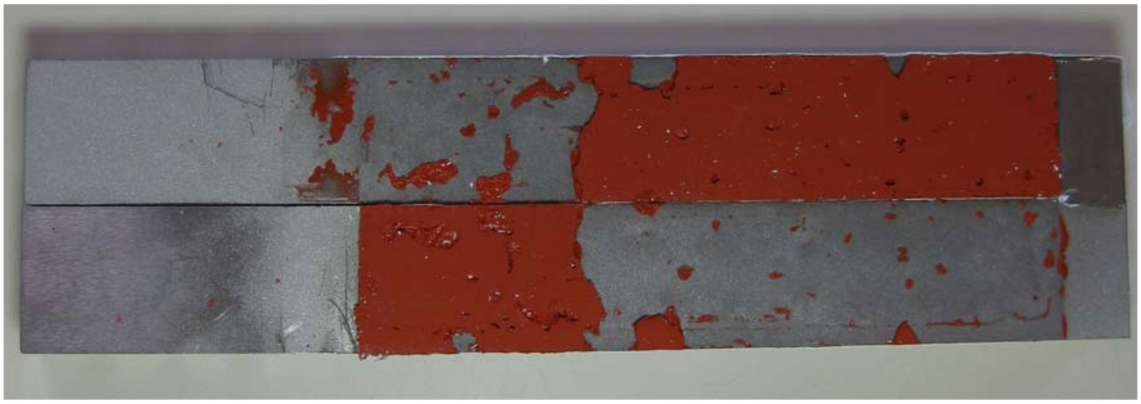


Figure 51 - Failure mode of DCB specimens

Chapter 5

5. Conclusions

In this thesis, a high temperature adhesive RTV 106 was studied through adhesive joint tests. The following conclusions can be drawn:

- The standard test TAST was performed in order to measure the shear properties of the adhesive.

- SLJs were tested at RT. From the tests results the lap shear strength of the adhesive is calculated. The average lap shear strength is comparable to the shear strength gives by the TAST.

- DCB test were performed in order to obtain the toughness for RTV 106. The toughness obtained is comparable to strong and brittle epoxy adhesives.

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ANNEXES 1

Standard Test Methods – ASTM test

The testing of adhesives and sealants is a complex subject because there are many parameters that can affect the test result.

ASTM's adhesives standards are helpful in the evaluation and testing of adhesives. Standard tests, such as ASTM tests, are very useful for comparing and determining the consistency of materials and processes.

Adhesives standards allow various companies worldwide to test adhesives and their components. These adhesives can have different properties depending on their volatile and non-volatile contents. The standards help to identify these properties which include viscosity, adhesion, shear strength, and shear modulus. The standards also help to identify adhesive bond or joint mechanical properties which include strength, creep, fracture, and fatigue. The different effects of environmental factors on adhesive bonds and joints are also evaluated. The adhesives standards are instrumental in determining various applications of adhesives such as in electrical insulation, sealing materials, and materials used in lumber products and floor systems.

There are two general categories of tests for adhesives and sealants: fundamental property tests and end-use tests. Fundamental tests can be used for monitoring the consistency of incoming products that are used in the adhesive bonding or sealing operations. End-use tests, are those which try to simulate the type of loading and service conditions to which a joint will be subjected.

3.2 Classification by substrate type of ASTM global standard test methods

Wood Adhesives [8]

a.) ***ASTM D905-03 Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading***

This test method covers the determination of the comparative shear strengths of adhesive bonds used for bonding wood and other similar materials, when tested on a standard specimen under specified conditions of preparation, conditioning, and loading in compression. This test method is intended primarily as an evaluation of adhesives for wood.

The values stated in SI units are to be regarded as the standard. The values given in brackets are provided for information purposes only.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

b.) ***ASTM D906-98 (2004) Standard Test Method for Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading***

This test method covers the determination of the comparative shear strengths of adhesives in plywood-type construction, when tested on a standard specimen and under specified conditions of preparation, conditioning, and testing. This test method is intended to be applied only to adhesives used in bonding wood to wood.

The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

c.) *ASTM D1101- 97a (2006) Standard Test Methods for Integrity of Adhesive Joints in Structural Laminated Wood Products for Exterior Use*

The test is suitable for check testing properly selected samples from regular production or for determining the suitability of adhesives and bonding techniques for the production of joints for exterior service. Vary the number of specimens taken from each member and the number of members selected for test depending on the total number of members involved and the purpose of the tests. It is advisable, however, to include at least three specimens in the test, with the specimens selected to represent the probable range of quality of the adhesive joints.

d.) ASTM D2339- 98 (2004) Standard Test Method for Strength Properties of Adhesives in Two-Ply Wood Construction in Shear by Tension Loading

This test method covers the determination of the comparative shear strengths of adhesives when tested on a standard specimen and under specified conditions of preparation, conditioning, and testing. This test method is intended to be applied only to adhesives used in bonding wood to wood.

e.) ***ASTM D3535-07a Standard Test Method for Resistance to Creep Under Static Loading for Structural Wood Laminating Adhesives Used Under Exterior Exposure Conditions***

This test method covers adhesives suitable for the bonding of wood, including treated wood, into structural laminated wood products for general construction, for marine use, or for other uses where a high-strength general construction, creep-resistant, waterproof adhesive bond is required.

f.) ***ASTM D4688-99 (2005) Standard Test Method for Evaluating Structural Adhesives for Finger Jointing Lumber***

This test method is specifically designed to measure the performance of adhesives in finger joints manufactured under production line conditions.

The results of the test method may be used to certify an adhesive as suitable for finger jointing lumber under production-line conditions where the intended end use of the finger jointed lumber will be in a structural glued laminated timber. When the test results are to be used for certification of an adhesive for this purpose, use a standard wood species and a standard finger profile.

g.) ***ASTM D5824-98 (2003) Standard Test Method for Determining Resistance to Delamination of Adhesive Bonds in Overlay-Wood Core Laminates Exposed to Heat and Water***

This test method provides a procedure to determine the quality of bond between an overlay and a wood core in an adhesively bonded laminate. The quality of bond is determined by measuring the resistance to delamination of the adhesively bonded laminate when tested under specific conditions of preparation, conditioning, and testing. Such products include, but are not limited to, window and door components, such as stiles and rails, and other overlaid panels. Typical wood-based cores are finger-jointed lumber, particleboard, oriented strand board, and hardboard. Typical overlays would be veneer, high-pressure laminate, high-density polyethylene, and fiberglass-reinforced plastic. Adhesive bond performance as measured by resistance to delamination in this test method is suitable for use in adhesive product development, manufacturing quality control, and monitoring bonding processes.

h.) ***ASTM D7247- 07ae1 Standard Test Method for Evaluating the Shear Strength of Adhesive Bonds in Laminated Wood Products at Elevated Temperatures***

This test method provides a procedure to determine the quality of bond between an overlay and a wood core in an adhesively bonded laminate. The quality of bond is determined by measuring the resistance to delamination of the adhesively bonded laminate when tested under specific conditions of preparation, conditioning, and testing. Such products include, but are not limited to, window and door components, such as stiles and rails, and other overlaid panels. Typical wood-based cores are finger-jointed lumber, particleboard, oriented strand board, and hardboard. Typical overlays would be veneer, high-pressure laminate, high-density polyethylene, and fiberglass-reinforced plastic. Adhesive bond performance as measured by resistance to delamination in this test method is suitable for use in adhesive product development, manufacturing quality control, and monitoring bonding processes. This test method is intended for the evaluation of adhesives that can be used to assemble test specimens in accordance with Test Method D905. The evaluation of other types of adhesives, such as the binder systems used for strand-based products, is beyond the scope of this test method. This test method is intended for the evaluation of adhesives as a component of laminated wood products at elevated temperatures. The evaluation of fire performance on fire-rated laminated wood products or assemblies is beyond the scope of this test method.

Metal Adhesives

a.) ***ASTM D897-01e1 Standard Test Method for Tensile Properties of Adhesive Bonds***

This test method covers the determination of the comparative tensile properties of the adhesive bonds of metal to metal when tested on standard shape specimens and under defined conditions of pretreatment, temperature, and testing machine speed.

b***.) ASTM D903-98 (2004) Standard Test Method for Peel or Stripping Strength of Adhesive Bonds***

This test method covers the determination of the comparative peel or stripping characteristics of adhesive bonds when tested on standard-sized specimens and under defined conditions of pretreatment, temperature, and testing machine speed.

c.) ***ASTM D950-03 Standard Test Method for Impact Strength of Adhesive Bonds***

Adhesives can fail under a sudden impact load and not under a slowly applied load of the same or greater force. This test method can be used to compare the sensitivity of various adhesives to suddenly applied loads. This test method covers the determination of the comparative impact strength of adhesive bonds in shear, when tested on standard specimens under specified conditions of preparation, conditioning, and testing.

d.) ***ASTM D1002-05 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)***

This test method covers the determination of the apparent shear strengths of adhesives for bonding metals when tested on a standard single-lap-joint specimen and under specified conditions of preparation and test. This test method is primarily comparative. However, it does have application as a discriminator in determining variations in adherend surface preparation parameters and adhesive environmental durability. The test method has found applications in controlling surface preparations, primer, and adhesive systems for determining strength properties of tested systems.

e.) ***ASTM D1062-02 Standard Test Method for Cleavage Strength of Metal-to-Metal Adhesive Bonds***

This test method covers the determination of the comparative cleavage properties of adhesive bonds when tested on standard shape metal specimens under specified conditions of preparation and testing. It may be used also to compare adhesives used with other metallic materials having any specified surface treatment.

f.) ***ASTM D1184-98 (2004) Standard Test Method for Flexural Strength of Adhesive Bonded Laminated Assemblies***

This test method covers the determination of the comparative properties of either metal or wood adhesive bonded assemblies when subjected to flexural stresses with standard shape specimens and under defined conditions of pretreatment, temperature, relative humidity, and testing technique. The test specimen and testing technique were designed to develop a large portion of shear forces between the laminae of the test piece when the load is applied, rather than to reduce shear stress to a minimum as is done in other ASTM test methods for flexural properties. This method is not applicable to assemblies made with nonrigid adherends. The data obtained are not suitable for design work.

g.) *ASTM D2293-96 (2008) Standard Test Method for Creep Properties of Adhesives in Shear by Compression Loading (Metal-to-Metal)*

This test method is useful in research and development for comparison of creep properties of adhesives, particularly as those properties are affected by changes in adhesive formulation or expected service conditions, including temperature, moisture level, and duration of loading. This test method covers the determination of the creep properties of adhesives for bonding metals when tested on a standard specimen and subjected to certain conditions of temperature and compressive stress in a spring-loaded testing apparatus. The relative size and simplicity of design of the spring-loaded apparatus permits easy portability and transfer from one environment to the next without disturbing static loads. The relative simplicity of design with inexpensive materials permits replication of creep tests at relatively low costs.