

Determination of the toughness in mode II for different types of adhesive

Daniel Ghibirgiu

Final project in MIEM

Supervisor: Prof. Lucas.F.M.da Silva

Co – Supervisor: Eng. Filipe José Palhares Chaves



University of Porto
Faculty of Engineering



“Dunarea de Jos” University of Galati
Faculty of Mechanical Engineering

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Abstract

In this thesis the toughness in mode II for three types of adhesives with 1 millimeter layer thickness is determined.

A very ductile adhesive SIKAFLEX 255, a brittle adhesive AV138/HV998 and an intermediate adhesive Araldite 2015 were used. The substrates were of heat treated steel with a thickness of 12, 7 mm as referred by the ASTM D3433 for the DCB specimen. The test method used was the ENF (End Notched Flexure), an easy test and widely used.

The experimental study is supported by an extensive literature review on adhesive bonding and fracture mechanics.

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List of Abbreviations

ASTM	American Society for testing and Materials
CDCB	Contoured double-cantilever beam
CLS	Cracked lap shear
DCB	Double Cantilever Beam
EDT	Edge delimitation tension
ELS	End Load Split
ENF	End Notched Flexure
FPZ	Fracture process zone
MMB	Mixed mode bending
PSA	Pressure sensitive adhesives
RTV	Room Temperature Vulcanizing
SLJ	Single Lap Joint
TDCB	Tapered Double Cantilever Beam

Glossary

Adhesive - a material which is adhesive resistant and applicable as a no sticking surface coating; release agent

Adherend - a body which is held to another body by an adhesive.

Bond line - the layer of adhesive which attaches two adherends.

Catalyst - a substance that markedly speeds up the cure of an adhesive when added in minor quantity as compared to the amounts of the primary reactants.

Cohesive Failure - Condition of bond failure in which the adhesive falls apart.

Cure - to change the physical properties of an adhesive by chemical reaction, which may be condensation, polymerization, or vulcanization; usually accomplished by the action of heat and catalyst, alone or in combination with or without pressure.

Delamination - the separation of layers in a laminate because of failure of the adhesive, either in the adhesive itself or at the interface between the adhesive and the adherend.

Degrease - to remove oil and grease from adherent surfaces.

Gap Fill - Ability of an adhesive to fill the space between substrates and hold the substrates in place.

Joint - the location at which two adherends are held together with a layer of adhesive.

Primer - a coating applied to a surface, prior to application of an adhesive, to improve performance of the bond.

Release agent - an adhesive material which prevents bond formation.

Sealant - a gap filling material to prevent excessive absorption of adhesive, or penetration of liquid or gaseous substances.

Temperature, ambient - temperature of the air surrounding the object under construction or test.

Young's Modulus – It is a mechanical propriety that is characteristic of each material and relates to its elastic behavior.

Notation

T	Temperature
E	Young's modulus
k	Bending moment factor
G	Shear modulus
P	Applied load
f	Natural frequency
l	Length
M	Bending moment on the adherent at the end of the overlap
t	Thickness
δ	Loss angle
ε	Tensile strain
γ	Shear strain
K	Stress intensity factor
σ	Tensile stress
σ_y	Tensile yield stress
τ	Shear stress
ρ	Density
ν	Poisson's ratio
λ	Constant function of adhesive and adherent elastic properties
ξ	Non-dimensional coordinate along the adhesive thickness.
I	Second moment of area
j	Geometric factor
Q	Constant function of geometry and loading

INTRODUCTION

1.1 Historical background

Adhesion is a concept present in humankind life since the very beginnings. While studying pre-historical tribal graveyards, archeologists discovered near skeletons, clay pottery fragments glued together with resins from tree sap. Ancient Babylon temple statues with eye globes glued to eye cavities were also discovered with some kind of tar resisting to 6000 years weathering degradation.

A good example of early adhesive use is the Ícarus legend and his wings built by his father Dedalus with feathers glued together with wax that melted when Ícarus got dangerously near to the Sun.

Paintings and murals from 1500 to 1000 B.C. figure wood gluing. In the Egyptian civilization animal origin glues were used to seal the tombs. However the first bibliographic reference about adhesives and the art of adhesion dates to 200 B.C.

Circa 1 and 1500 A.C., the Greeks and Romans developed ornamentation technique by gluing small noble wood layers. To do so, they used animal origin glues, mostly fish glue, egg whites, blood, bones, milk, cheese and some vegetables glue. In naval industry Romans used bee wax and tar in order to joint wood segments and to obtain waterproof decks.

During the following centuries gluing was a standard procedure in almost every object, from furniture to Stradivarius violins. However it was only in the eighteenth century (circa 1700) that the first industrial glue factory was settled in the Netherlands.

In England, the first patent attributed to an adhesive was about 1750, and consisted of fish glue. The following patents were for natural rubber, milk,

casein and gum glues. In the early years of the twenty century, there were innumerable glue factories all over the World. Throughout the First World War, casein adhesives were used to build wood airplanes and by the Second World War modified phenolic resins were used to promote structural connection, once again in the aeronautic industry.

Despite the fact that the knowledge and common use of adhesives remounts to early ages (4000 B.C.), it is just after the twentieth century, when polymers were developed following the industrial expansion, that we observe a sustainable, science supported, and technically based adhesive developments, enriching the knowledge database. This development was also due to the availability of a great variety of formulations improving the adhesion performance of the adhesives, by controlling mechanical properties, curing time, service temperatures and chemical resistance. A good example is the plywood industry that boosted the development of phenolic adhesives and phenolic bakelite [1].

In medicine, adhesives are used in substitution of common scar sewing to close wounds with cyanoacrylate. In dental restoration, light and UV adhesives and sealants are used.

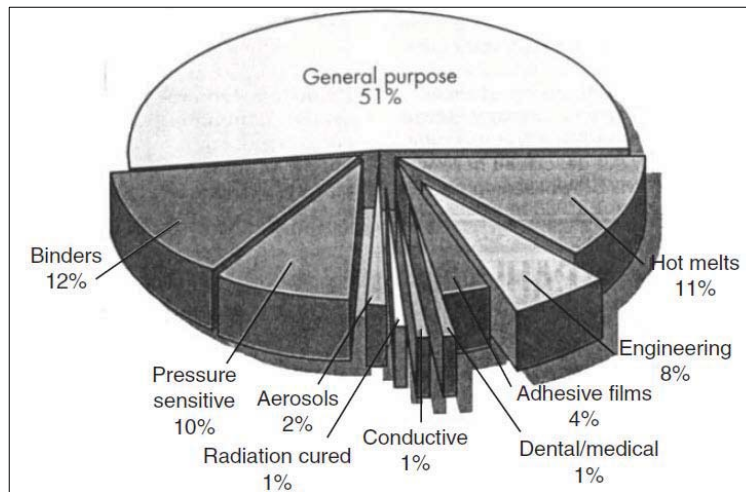
The development of modern polymeric adhesives and sealants began about the same time as the polymer industry itself, early in the 1900s. In fact, the polymeric and elastomeric resins industry is bound very closely to the adhesive and sealant industries.

Adhesives and sealants found important markets in the construction industry, which was providing much of the growing infrastructure in the U.S. at the time.

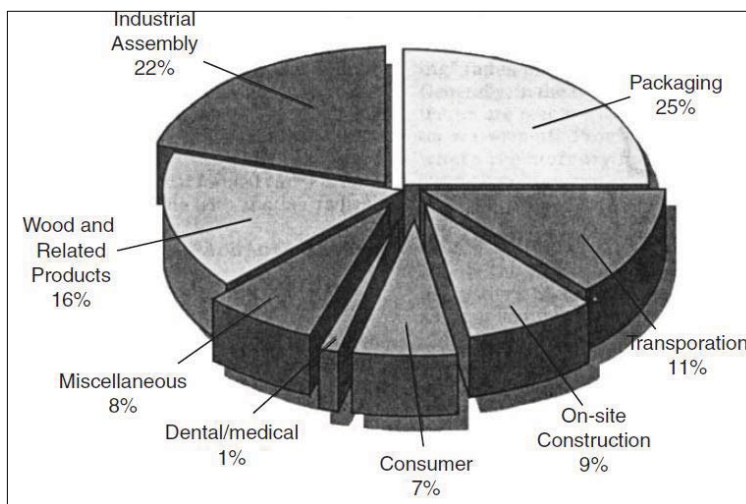
1.2 Adhesive market

In 1995 the adhesive market represented about 26 billion dollars and was growing 3% every year (8-9% in emergent Asiatic markets).

The market segmentation is represented in the following figures [2]. General purpose adhesives represent the major part of the market (51%), followed by binders, hot melts and engineering adhesives. Figure 1 shows that “packaging” and industrial assembly is the two leading end-use markets for adhesives, followed closely by wood and its related products.



(a)



(b)

Figure 1. Leading adhesive and sealant (a) products and (b) end-use markets in 1995 [3].

Geographically, the market distribution was analyzed in 2000 by the ChemQuest Group and is distributed as shown in Figure 2.

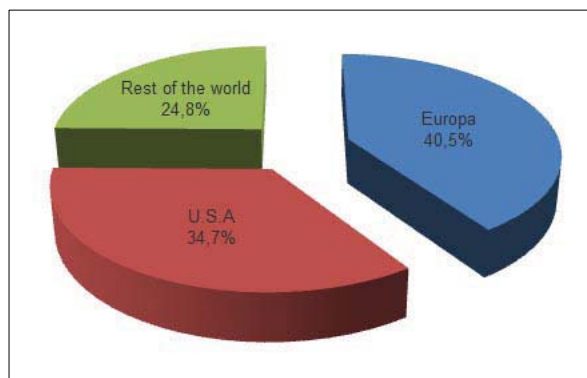


Figure 2. World adhesive markets.

Europe and U.S.A are the two leading markets for adhesives. Recently the Asian market is becoming more relevant because of the Chinese industrial booming.

1.3 Importance of adhesives and sealants

Adhesives and sealants surround us in nature and in our daily lives. Substantial businesses exist to develop, manufacture, and market these materials, and they are used within virtually every business and industry. Applications abound from office “post-it notes” to automotive safety glass to footwear to aerospace structures to “no-lick” postage stamps [2]. Many products that we take for granted could never exist if it were not for adhesive bonding or sealing.

Adhesives and sealants industry is bolstered by thousands of years of trial and error. This long history can be coupled with significant additions to the fundamental supporting sciences and with the development of advanced materials and processes. Consequently, society has generally progressed to a point where we actually trust not only our fortunes but also our lives to these

materials. The study of adhesives and sealants and the sciences surrounding their application has never been more important [2].

1.4 Definitions

Like any science, which has evolved over the centuries, the science that supports sealants and adhesives developed a jargon and language of its own. Importantly, the basic terms needed to develop an understanding of how and why adhesives and sealants provide value are presented in this section.

Adhesive - a substance, such as paste or cement, that causes two surfaces to stick together. Adhesive or glue is a compound in a liquid or semi-liquid state that adheres or bonds items together.

Adhesives may come from either natural or synthetic sources. Some modern adhesives are extremely strong, and are becoming increasingly important in modern construction and industry. The types of materials that can be bonded using adhesives are virtually limitless, but they are especially useful for bonding thin materials. Adhesives usually require a controlled temperature and temperature to cure or set. They can be electrically and thermally conductive or nonconductive.

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.

No matter what the application, a sealant has three basic functions:

- It fills a gap between two or more substrates;
- It forms a barrier through the physical properties of the sealant itself and by adhesion to the substrate;
- It maintains sealing properties for the expected lifetime, service conditions and environments.

Both adhesives and sealants function primarily by the property of adhesion.

Adhesion is the attraction of two different substances resulting from intermolecular forces between the substances. This is distinctly different from *cohesion*, which involves only the intermolecular attractive forces within a single substance.

The main difference between adhesives and sealants is that sealants typically have lower strength and higher elongation than do adhesives. Since the main objective of a sealant is to seal assemblies and joints, sealants need to have sufficient adhesion to the substrates and resistance to environmental conditions to remain bonded over the required life of the assembly. When sealants are used between substrates having different thermal coefficients of expansion or differing elongation under stress, they need to have adequate flexibility and elongation. Sealants generally contain inert filler material and are usually formulated with an elastomer to give the required flexibility and elongation. They usually have a paste consistency to allow filling of gaps between substrates.

Structural adhesive is a term generally used to define an adhesive whose strength is critical to the success of the assembly. This term is usually reserved to describe adhesives with high shear strength (in excess of 1,000 pounds per square inch or psi) and good environmental resistance.

Non-structural adhesives are adhesives with much lower strength and permanence. They are generally used for temporary attachment or bond to weak substrates.

An important definition may be illustrated by considering the schematic of the joint in Figure 3 where two substrates are bonded together with an adhesive or sealant. The *substrate* is the material to be bonded. After bonding, the substrate is often referred to as an adherent.

Sometimes a *primer* is used with adhesives or sealants. A primer is applied to a surface prior to the application of an adhesive or sealant, usually for

improving the performance of the bond or protecting the surface until the adhesive or sealant can be applied.

The *joint* is the part of the assembly made up of adherends; adhesive or sealant; primers, if present; and all associated interphase regions as shown in Figure 3.

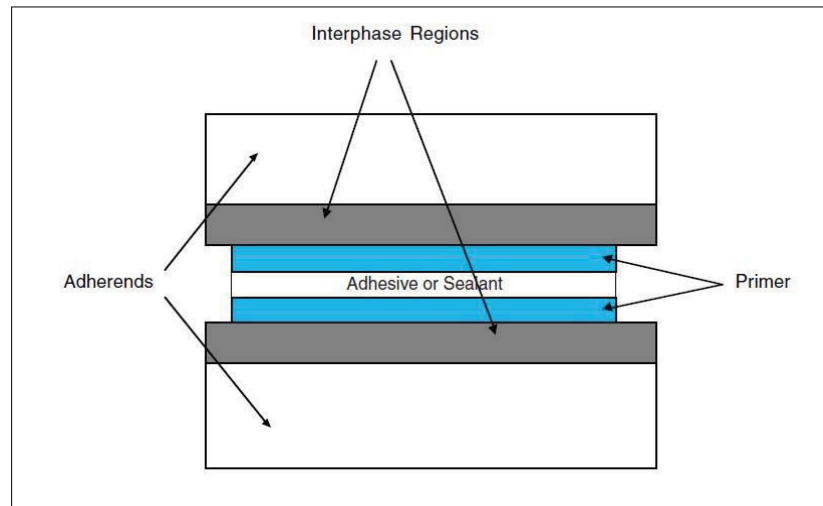


Figure 3. Components of a typical adhesive or sealant joint [2].

1.5 Selection and classification of adhesives

The properties of a bonded joint are strongly affected by the choice of adhesive. Different types of adhesives have different properties. The choice should be made in consultation with manufacturers or specialists in adhesive technology. It is very important to provide as complete information on the properties required as possible. The choice of adhesive depends on a lot of factors such as:

- processing requirements;
- material to be bonded;
- strength requirements;
- service conditions;
- type of assembly;

- health aspects;
- service life;
- costs.

Science of adhesion is supported by knowledge of several disciplines. It is really a wide range of exploration in search of better solutions. Multidisciplinary aspects of integration are reflected in Figure 4, where science of adhesion assumes central position as a result of integration other disciplines.

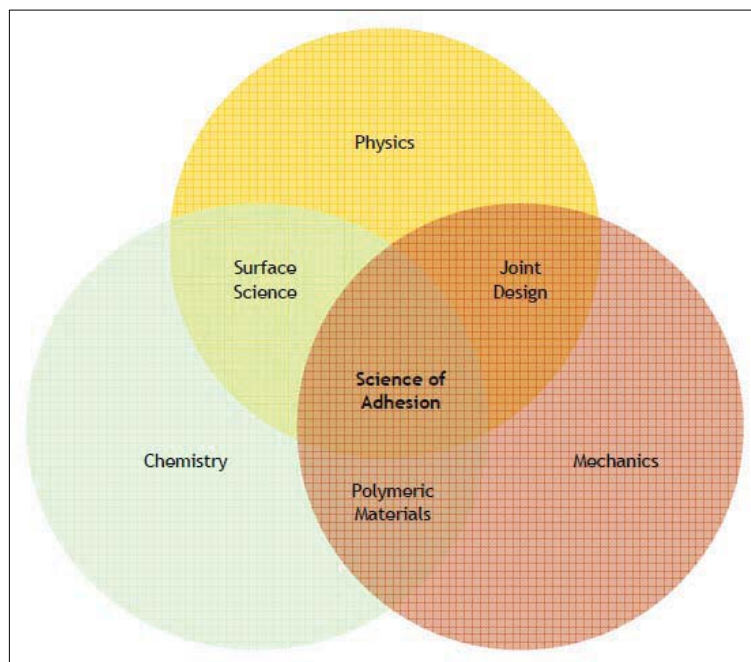


Figure 4. Science of adhesion; Multidisciplinary graph [1].

Adhesives are classified by different classification systems.

General classification of adhesives (classification by structure)

Thermosetting adhesives - Thermosets molecules are cross-linked by strong covalent intermolecular bonds, forming one giant molecule. Cross-linking is irreversible therefore thermosets can't be reprocessed (re-melt). Cross-linking is achieved in curing process initiated by heat, chemical agents, radiation or

evaporation of solvents. Curing results in sharp increase of strength, elasticity and stability of thermosets.

Thermoplastic adhesives - Thermoplastics are polymers, which soften (become pliable and plastic) and melt when heated. No new cross-links form (no chemical curing) when a thermoplastic cools and harden. Thermoplastics may be reprocessed many times by heating or applying a solvent. Molecules of most of thermoplastics combine long polymer chains alternating with monomer units.

Elastomeric adhesives - Elastomers are polymers possessing high elasticity and which may be reversibly stretched at high degree. Elastomers consist of long lightly cross-linked molecules. Elastomers are set (strengthened) by thermal curing or solvent evaporation. Curing results in increase of cross-linking of the molecules.

Hybrid adhesives – made by combining thermosetting, thermoplastic and elastomeric resins into a single adhesive formulation.

Classification of adhesives by curing method

One-part adhesives

- ***Heat activated curing adhesives.*** Adhesives of this type (epoxies, urethanes, and polyimide) 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 photo initiators enhancing curing reaction.
- ***Moisture activated curing adhesives (RTV silicones, cyanoacrylates, urethanes).*** These adhesives are cured when react with a moisture present on the substrate surface or in the air. ***RTV silicones adhesives*** are a rubber-like polymer called polydimethylsiloxanes. RTV stands for “room

temperature vulcanizing” or simply a rubber that cures at room temperature. *Cyanoacrylates* are extremely rapid curing adhesives commonly called “superglue”. Cyanoacrylates are typically used in applications where there is a need for a rapid-curing, single-component adhesive that provides high adhesion, high tensile strength, and easy dispensing.

- *Anaerobic adhesives* – are derived from methacrylates, a monomer related to acrylics or more commonly known as plexiglass. The term anaerobic is used to describe this family of adhesives because this type of adhesive “comes to life” or hardens in the absence of air. There are many different types of anaerobics used for specific applications such as threadlocking, threadsealing, flangesealing or retaining.
- *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.

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. **Epoxies** are adhesive systems made by a complex chemical reaction. Various resins are made synthetically by reacting two or more chemicals. The resultant resin can then be reacted or cured by the addition of another chemical called a hardener, or catalyst. Epoxy adhesives can bond a wide variety of substrates (particularly metals) with high strength. They have been used to replace some traditional metalworking methods of joining like nuts and bolts, rivets, welding, crimping, brazing, and soldering.

Classification of adhesives by origin

Synthetic adhesives - Adhesives based on synthetic resins and rubbers excel in versatility and performance. Synthetics can be produced in a constant supply and at constantly uniform properties. In addition, they can be modified in many ways and are often combined to obtain the best characteristics for a particular application.

Natural adhesives - Generally set by solvent evaporation. They are generally of low strength and are susceptible to moisture and mould. Their use is restricted to the joining of low strength materials. Natural adhesives include animal glues, fish glues, vegetable glues and casein.

- ***Animal glue*** is made from the protein extracted from the bones, hide, hoofs, and horns of animals by boiling. The extract is cooked to form a gelatin material. The gelatin can then be re-liquefied with heat, which gives it quick setting properties. Its major use has been in the wood and furniture industry. If you have seen a heated glue pot with a brush in it, it was probably animal glue. Animal byproducts from meat processing have been the source of supply for this type of glue as well as the sources of jokes about old Dobbin being past his prime and only good for the “glue pot”.
- ***Vegetable glue*** is a carbohydrate extracted from vegetable plants such as corn, rice, wheat, potatoes, and is probably better known as paste. Major use area is in bonding paper and paper products such as bookbinding, corrugated boxes, paper bags, and wallpaper paste (no removable); it is also used as a sizing in textiles. The laundry uses starch on your shirt collars to stiffen and give shape to your shirt.
- ***Fish glue*** is similar protein-based glue made from the skins and bones of fish. An exceptionally clear adhesive can be made from fish and was the

first adhesive used for photographic emulsions for photo film and photo resist coatings for photoengraving processes.

- **Casein Glue** is made from a protein isolated from milk. The extraction process creates an adhesive that is waterproof. Its first use was in bonding the seam of cigarette paper. It provides a fast-setting bond that requires very little adhesive; one gram of adhesive can bond 2,000 cigarettes.

1.6 Reaction principles of the adhesive

The transmission of the force in an adhesive bond is caused by the interaction between adhesion and cohesion forces. The forces acting between the adhesive and the molecules in the mating component are defined as adhesion, and the forces acting between the individual adhesive molecules are defined as cohesion.

Adhesion - describes the regularity of the adhesion between the adhesive layers with the surface of the mating component; adhesion is the tendency of certain dissimilar molecules to cling together due to attractive forces.

Mechanisms of adhesion

Mechanical adhesion - Adhesive materials fill the voids or pores of the surfaces and hold surfaces together by interlocking. Sewing forms a large scale mechanical bond, Velcro forms one on a medium scale, and some textile adhesives form one at a small scale. This is similar to surface tension.

Chemical adhesion - Two materials may form a compound at the joint. The strongest joins are where atoms of the two materials swap (ionic bonding) or share (covalent bonding) outer electrons. A weaker bond is formed if oxygen, nitrogen or fluorine atoms of the two materials share a hydrogen nucleus (hydrogen bonding).

Dispersive adhesion - In dispersive adhesion, also known as adsorption, two materials are held together by van der Waals forces: the attraction between two

molecules, each of which has regions of positive and negative charge. In the simple case, such molecules are therefore polar with respect to average charge density, although in larger or more complex molecules, there may be multiple "poles" or regions of greater positive or negative charge. These positive and negative poles may be a permanent property of a molecule (Keesom forces) or a transient effect which can occur in any molecule, as the random movement of electrons within the molecules may result in a temporary concentration of electrons in one region (London forces). In surface science, the term "*adhesion*" almost always refers to dispersive adhesion. In a typical solid-liquid-gas system (such as a drop of liquid on a solid surrounded by air) the contact angle is used to quantify adhesiveness. In the cases where the contact angle is low, more adhesion is present. This is due to a larger surface area between the liquid and solid and results in higher surface energy. The Work of Adhesion explains the interactive force between the liquid and solid phases and the Young-Dupree equation is used to calculate the Work of Adhesion. The contact angle of the three-phase system is a function not only of dispersive adhesion (interaction between the molecules in the liquid and the molecules in the solid) but also cohesion (interaction between the liquid molecules themselves). Strong adhesion and weak cohesion results in a high degree of wetting, a lyophobic condition with low measured contact angles. Conversely, weak adhesion and strong cohesion results in lyophobic conditions with high measured contact angles and poor wetting.

Electrostatic adhesion - Some conducting materials may pass electrons to form a difference in electrical charge at the join. This results in a structure similar to a capacitor and creates an attractive electrostatic force between the materials.

Diffusive adhesion - Some materials may merge at the joint by diffusion. This may occur when the molecules of both materials are mobile and soluble in each other. This would be particularly effective with polymer chains where one end of the molecule diffuses into the other material. It is also the mechanism

involved in sintering. When metal or ceramic powders are pressed together and heated, atoms diffuse from one particle to the next. This joins the particles into one.

Cohesion – The term cohesion relates to the forces that determine the confinement of the adhesive. Cohesion is crucial to both the viscosity of a non-cured adhesive during processing and also to its strength after curing and under stress. We can use the following interactions to describe cohesion:

- the attraction of contiguous molecules and atoms owing to intermolecular and covalent forces;
- chemical bonds within molecule;
- the mechanical convoluting of filamentary molecules or fibrous constituents.

The cohesion of adhesive is characterized by key values such as Young's Modulus of elasticity, elongation at tear, shore hardness and temperature stability.

Surface tension - Surface tension is an attractive property of the surface of a liquid. It is what causes the surface portion of liquid to be attracted to another surface, such as that of another portion of liquid (as in connecting bits of water or as in a drop of mercury that forms a cohesive ball).

Surface-tension is caused by the attraction between the liquid's molecules by various intermolecular forces. In the bulk of the liquid, each molecule is pulled equally in all directions by neighboring liquid molecules (showed in Figure 5), resulting in a net force of zero.

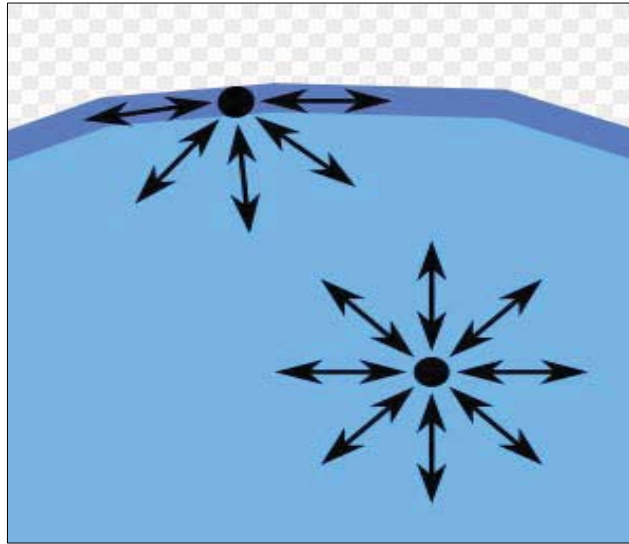


Figure 5. Diagram of the forces on two molecules of liquid [4].

At the surface of the liquid, the molecules are pulled inwards by other molecules deeper inside the liquid and are not attracted as intensely by the molecules in the neighboring medium (be it vacuum, air or another liquid). Therefore, all of the molecules at the surface are subject to an inward force of molecular attraction which is balanced only by the liquid's resistance to compression, meaning there is no net inward force. However, there is a driving force to diminish the surface area, and in this respect a liquid surface resembles a stretched elastic membrane. Thus the liquid squeezes itself together until it has the locally lowest surface area possible.

Surface tension, represented by the symbol γ is defined as the force along a line of unit length, where the force is parallel to the surface but perpendicular to the line.

One way to picture this is to imagine a flat soap film bounded on one side by a taut thread of length, L as shown in Figure 6.

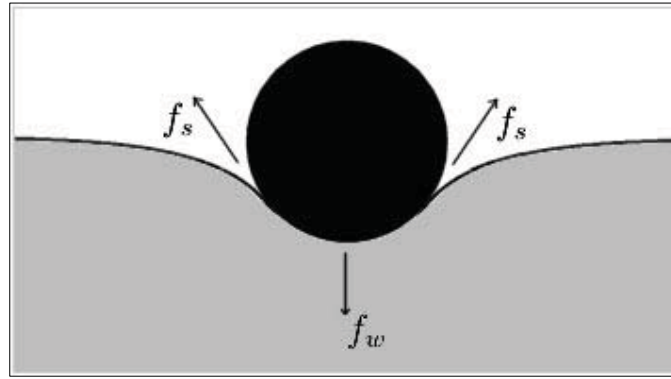


Figure 6. Diagram shows, in cross-section, a needle floating on the surface of water [5].

Its weight, f_w , depresses the surface, and is balanced by the surface tension forces on either side, f_s , which are each parallel to the water's surface at the points where it contacts the needle. Notice that the horizontal components of the two f_s arrows point in opposite directions, so they cancel each other, but the vertical components point in the same direction and therefore add up to balance f_w .

Surface energy - Surface energy quantifies the disruption of intermolecular bonds that occurs when a surface is created. In the physics of solids, surfaces must be intrinsically less energetically favorable than the bulk of a material; otherwise there would be a driving force for surfaces to be created, and surface is all there would be the surface energy may therefore be defined as the excess energy at the surface of a material compared to the bulk.

Cutting a solid body into pieces disrupts its bonds, and therefore consumes energy. If the cutting is done reversibly then conservation of energy means that the energy consumed by the cutting process will be equal to the energy inherent in the two new surfaces created. The unit surface energy of a material would therefore be half of its energy of cohesion, all other things being equal; in practice; this is true only for a surface freshly prepared in vacuum. Surfaces often change their form away from the simple "cleaved bond" model

just implied above. They are found to be highly dynamic regions, which readily rearrange or react, so that energy is often reduced by such processes as passivation or adsorption.

1.7 Important factors for successfully using adhesives and sealants

Adhesion is the principal factors in the success of either an adhesive or sealant. In the following we make classification criteria for several of the factors influencing the adhesives and sealants [2].

Chemical Factors:

- external - effect of chemical agents such as water, gasoline, by hydraulic fluid, acids, alkalies;
- internal - effect of adherent on adhesive ;effect of adhesive on the adherent

Exposure:

- weathering – combined effect of rainfall, sunlight, temperature changes, type of atmosphere;
- light - important only with translucent adherents;
- oxidation - tested by exposure to ozone , in which case deterioration is faster;
- moisture - excessive moisture is usually undesirable because can cause corrosion in metals;
- salt spray - important only in coastal or marine atmospheres.

Temperature:

- high - bond strength may be affected by reactions in adhesive or adherent at a normal atmospheric variations or exceptional conditions;
- low - low temperatures may cause crystallization or embrittlement.

Working Properties:

- application - consistency or viscosity may be adequate indications. Mechanical stability of emulsions and dispersions, and foaming tendency, can be important for machine application.
- bonding range - Minimum drying or solvent-reactivation time before suitable bond can be obtained. Maximum allowable time before assembly.
- curing rate - Minimum curing time, and effect of over curing. May be determined as a shear or tensile-strength vs. curing-time curve at a specific curing temperature.

1.8 Advantages and disadvantages of adhesive bonding

As well as other joining methods, adhesive bonding has some advantages and disadvantages. The result is strongly dependent on joint design, choice of adhesive, surface preparation and the bonding process.

Advantages of adhesive bonding are:

- uniform stress distribution over the entire bonded area;
- good fatigue resistance;
- ability to join dissimilar materials;
- fast and cheap joining technique
- uniform distribution of mechanical stress over the joint;
- good vibration-damping and flexibility;
- weight reduction compared to bolt and rivet joints;
- smooth joint surfaces and contours;
- ability to anodize with no risk of staining;
- prevention or reduction of galvanic corrosion;
- good sealing and insulating properties.

Disadvantages of adhesive bonding are:

- decreased strength at elevated temperatures, especially under long time loading;
- requires careful substrate (adherent) surface preparation;
- difficult disassembly of joined parts;
- necessity to fixture (hold together) the joined parts during curing;
- possible degradation of bond by environmental influence;
- complicated bonding process. Stronger joints demand proper surface preparation and may also require jigs, presses and heating equipment;
- difficult and expensive to provide for adequate inspection;
- additional exhaust and protective clothing might be necessary for reasons of health.

1.9 Joint design

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. There are characteristics of the adhesives which mean that they perform better in compression, shear and tension than in peel and cleavage. These forces are indicated in Figure 7

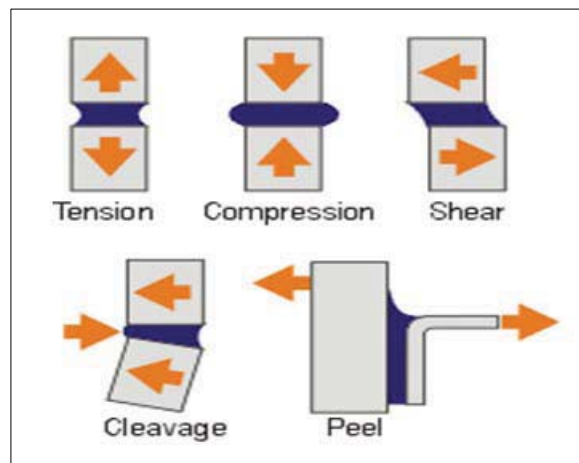


Figure 7. The types of load experienced by a joint [6].

Tensile stress (Tension) is exerted equally over the entire joint straight and away from the adhesive bond.

Shear stress is across the adhesive bond. The bonded materials are being forced to slide over each other.

Cleavage stress is concentrated at one edge and exerts a prying force on the bond.

Peel stress is concentrated along a thin line at the bond's edge. One surface is flexible.

When designing the joint it is necessary to:

- maximize tension, shear and compression forces;
- minimize peel and cleavage forces;
- optimize the area over which the load is distributed.

The strength of a joint is a complex function of the stress concentrations set up by the load. In a simple lap joint made from thin metal sheet there are two sorts of stress: shear and peel. The shear stress varies along the length of the joint with concentrations at the ends. The peel stress acts at right angles to the lap joint and is likewise at a maximum at the ends (Figure 8).

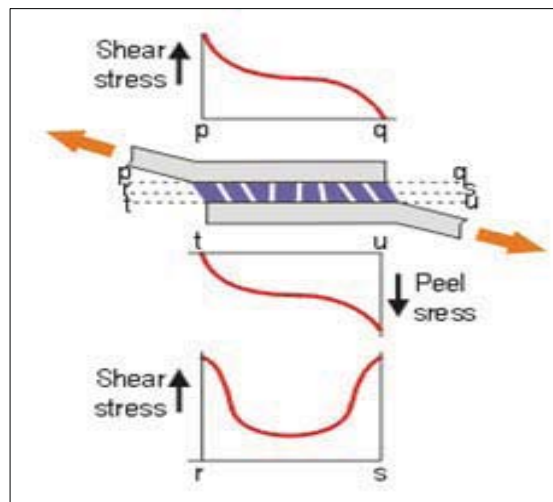


Figure 8. Stress distributions in a lap shear joint [6].

Alternative joint designs are presented in Figure 9. In most of these, the stresses are more evenly distributed. The efficiency gained results in joints of greater strength. These joints can be applied to more complex geometries including the stiffening of large thin sheets, strengthening around apertures, bonding of multilayer structures and joints using profiles.

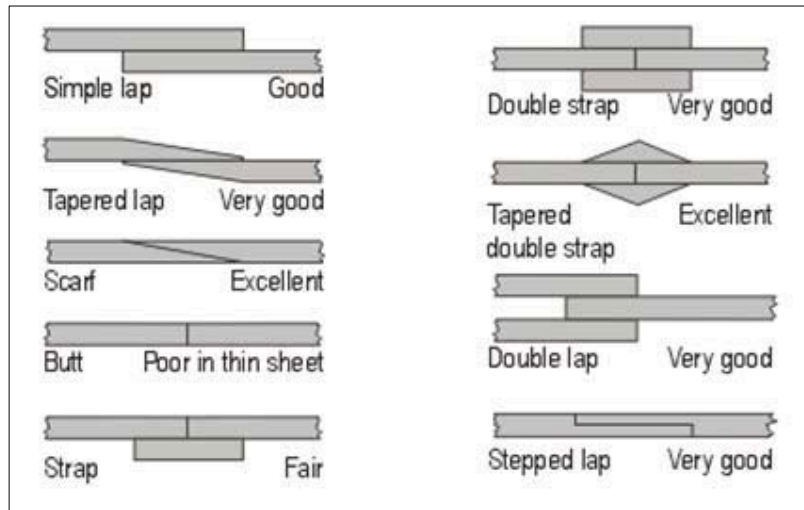


Figure 9. Basic joint geometries [7].

In practice, two or more may be used in conjunction with one another.

Lap joints are most common. They are most practical and applicable in bonding thin materials.

The simple lap joint is off-set when using thin materials. This can result in cleavage and peel stress under load.

A tapered single lap joint is more efficient than a simple lap joint. The tapered edge allows bending of the joint edge under stress.

The joggle lap joint gives more uniform stress distribution than either the simple lap or tapered lap joint. The joint can be formed by simple metal forming operations. The curing pressure is easily applied.

The double butt lap joint gives more uniform stress distribution in the load bearing area than the previously mentioned joints. This type of joint, however, requires machining which is not always feasible with thinner gauge metals.

Double scarf lap joints have better resistance to bending forces than double butt joints. This type of joint however also requires machining.

1.9.1 Other specific type's joints

- *Angle joints* give rise to either peel or cleavage stress depending on the gauge of metal. Typical approaches to the reduction of cleavage are illustrated in Figure 10.

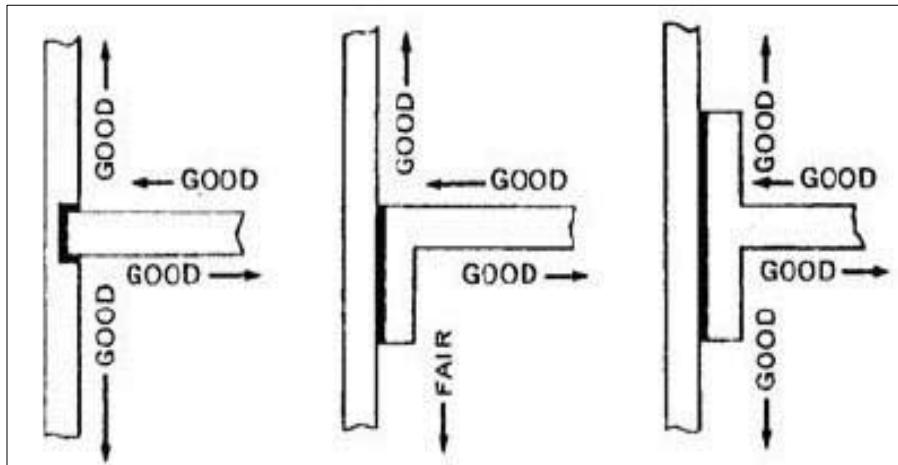


Figure 10. Typical approaches to the reduction of cleavage [8].

- *Butt joints* - A straight butt joint has poor resistance to cleavage. The following recessed butt joints are recommended: landed scarf tongue and groove, conventional tongue and groove, and scarf tongue and groove. Landed scarf tongue and groove joints act as stops which can control adhesive line thickness. Tongue and groove is self-aligning during assembly and act as a reservoir for mastic type void filling adhesives.

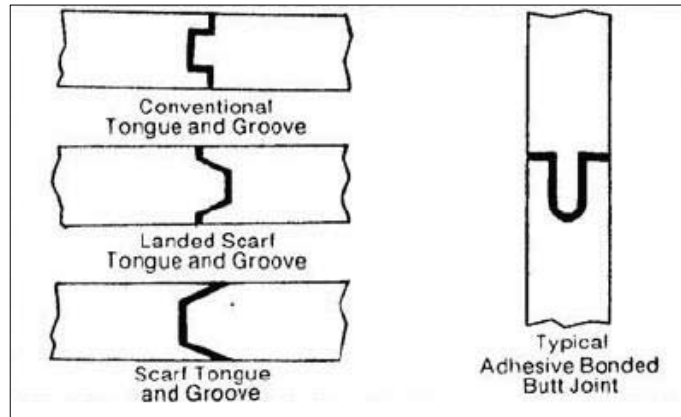


Figure 11. Butt joints [9].

- *Cylindrical joints* - The joint and overlap slip joint are typical for bonding cylindrical parts such as tubing, bushings, and shafts. With adhesive bonding, all available contact area contributes to carry the load. Adhesives also provide a joint with better appearance and eliminate distortion caused from high welding temperatures.

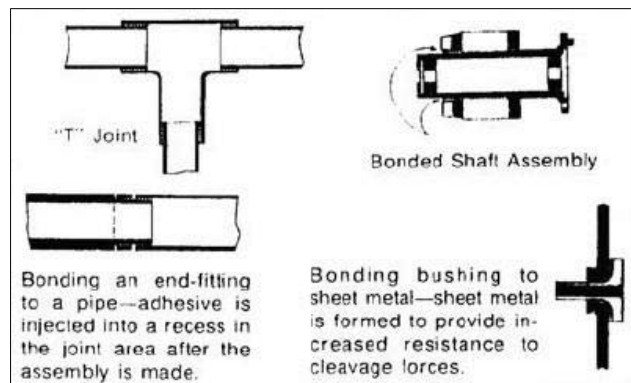


Figure 12. Cylindrical joints [10].

- *Corner joints — Sheet metal* - Corner joints can be assembled with adhesives by using simple supplementary attachments. This permits joint and sealing in a single operation. Typical designs are right angle butt joint, slip joints increase the structure's rigidity. Void filling adhesives are

most frequently used. Use of a heat curing adhesive depends on the heat resistance of the materials being bonded. With this technique, thin gauge metals or sandwich panels can be easily formed into boxes, case, housing, vehicle bodies, metal boat hulls, etc

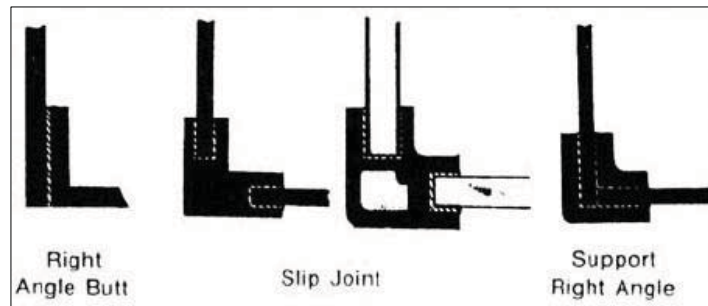


Figure 13. Sheet metal [11].

- *Corner joints — Rigid members* - Corner joints, as in storm doors or decorative frames, can be adhesive bonded. End lap joints are the simplest design type although they require machining. Adhesives requiring pressure during cure may be utilized in such designs. Mortise and tenon joints are excellent from a design standpoint but also require machining. Mitered joint with spline is best if members are hollow extrusions. In this case, a void filling adhesive is recommended.

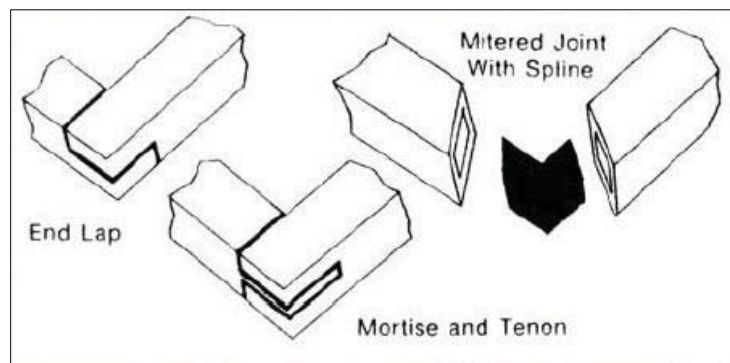


Figure 14. Rigid members [12].

1.9.2 Joint design considerations

There are several rules that the designer should consider when designing an adhesive joint [2], such as:

- keep the stress on the bond-line to a minimum;
- whenever possible, design the joint so that the operating loads will stress the adhesive in shear;
- peel and cleavage stresses should be minimized;
- distribute the stress as uniformly as possible over the entire bonded area;
- adhesive strength is directly proportional to bond width. Increasing width will always increase bond strength; increasing the depth of overlap does not always increase strength;
- generally, rigid adhesives are better in shear, and flexible adhesives are better in peel.
- although typically a stronger adhesive material may produce a stronger joint, a high elongation adhesive with a lower cohesive strength could produce a stronger joint in applications where the stress is distributed non uniformly.
- the stiffness of the adherents and adhesive influence the strength of a joint. In general, the stiffer the adherent with respect to the adhesive, the more uniform the stress distribution in the joint and the higher the bond strength.
- the higher the Et (modulus \times thickness) of the adherent, the less likely the deformation during load, and the stronger the joint.
- within reasonable limits, the adhesive bond-line thickness is not a strong influence on the strength of the joint. More important characteristics are a uniform joint thickness and void free adhesive layer.

FRACTURE MECHANICS

2.1 Introduction

Designs for engineering structures have been dominated by using a strength approach, in which the stresses (or in some cases strains) are compared to some allowable strength of the material. Stresses or strains may individually be compared to a limiting strength, or may be combined into an appropriate metric reflecting the effect of stress interactions, in methods such as the von Mises yield criterion, which has been widely applied to the design of ductile materials (Seely and Smith, 1952). Although such approaches have been widely and successfully applied, problems have arisen in a number of now infamous structural failures, in which pre-existing or service-induced flaws have propagated catastrophically, at times with significant loss of life and property (Broek, 1978). Since traditional strength-based approaches to design normally assume that the materials being used are continua, they are not well suited for applications to systems in which flaws, cracks, debonds, delaminations, damage, or other imperfections are present. Stresses and local strains are greatly increased at the tips of these defects, which often serve as initiation sites for structural failures. Historically, a more recent approach to analysis and design, fracture mechanics offers an alternative set of criteria for evaluating the integrity of real structures that may contain flaws, and is making important inroads into design [13].

Failures occur for several reasons, including uncertainties in the loading or environment, defects in the materials, inadequacies in design, and deficiencies in construction or maintenance. Design against fracture has a technology of its own, and this is a very active area of current research. The central difficulty in designing against fracture in high-strength materials is that

the presence of cracks can modify the local stresses to such an extent that the elastic stress analyses done so carefully by the designers are insufficient. When a crack reaches a certain critical length, it can propagate catastrophically through the structure, even though the gross stress is much less than would normally cause yield or failure in a tensile specimen. The term “fracture mechanics” refers to a vital specialization within solid mechanics in which the presence of a crack is assumed, and we wish to find quantitative relations between the crack length, the material’s inherent resistance to crack growth, and the stress at which the crack propagates at high speed to cause structural failure [14].

2.2 Fracture mechanics failure criteria

Fracture mechanics does not assume the material as continua, instead it considers imperfections such as a flaw, cracks, debonds and delamination, resulting of manufacture errors or in service loads. Other than local strain and stresses at crack tips, fracture mechanics evaluates if defects present in the material will lead to structural failure.

2.3 The energy-balance approach

When A.A. Griffith (1893–1963) began his pioneering studies of fracture in glass in the years just prior to 1920, he was aware of Inglis’ work in calculating the stress concentrations around elliptical holes, and naturally considered how it might be used in developing a fundamental approach to predicting fracture strengths. However, the Inglis solution poses a mathematical difficulty: in the limit of a perfectly sharp crack, the stresses approach infinity at the crack tip. This is obviously nonphysical (actually the material generally undergoes some local yielding to blunt the crack tip), and using such a result would predict that materials would have near zero strength: even for very small applied loads, the stresses near crack tips would become infinite, and the bonds

there would rupture. Rather than focusing on the crack-tip stresses directly, Griffith employed an energy-balance approach that has become one of the most famous developments in materials science [15].

The strain energy per unit volume of stressed material is:

$$U^* = \frac{1}{V} \int f dx = \int \frac{f}{A} \int \frac{dx}{L} = \int \sigma d \varepsilon \quad (1)$$

where,

V-volume

A-area

L-length

σ -strain

If the material is linear ($\sigma = E\varepsilon$), then the strain energy per unit volume is:

$$U^* = \frac{E\varepsilon^2}{2} = \frac{\sigma^2}{2E} \quad (2)$$

When a crack has grown into a solid to a depth a , a region of material adjacent to the free surfaces is unloaded, and its strain energy released. Using the Inglis solution, Griffith was able to compute just how much energy this is.

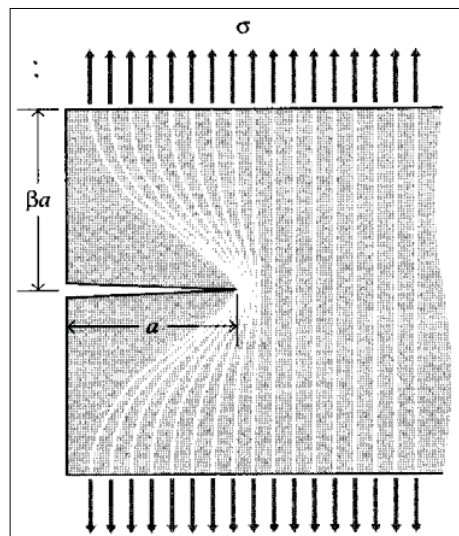


Figure 15. Idealization of unloaded region near crack flanks [15].

A simple way of visualizing this energy release, illustrated in Figure 15, is to regard two triangular regions near the crack flanks, of width a and height βa , as being completely unloaded, while the remaining material continues to feel the full stress σ .

The parameter β can be selected so as to agree with the Inglis solution, and it turns out that for plane stress loading $\beta = \pi$. The total strain energy U released is then the strain energy per unit volume times the volume in both triangular regions:

$$U = \frac{\sigma^2}{2E} \cdot \pi a^2 \quad (3)$$

Here the dimension normal to the x-y plane is taken to be unity, so U is the strain energy released per unit thickness of specimen. This strain energy is liberated by crack growth. But in forming the crack, bonds must be broken, and the requisite bond energy is in effect absorbed by the material. The surface energy S associated with a crack of length a (and unit depth) is:

$$S = 2\gamma a \quad (4)$$

where γ is the surface energy (e.g., J/m^2) and the factor 2 is needed since two free surfaces have been formed. As shown in Figure 16, the total energy associated with the crack is then the sum of the (positive) energy absorbed to create the new surfaces, plus the (negative) strain energy liberated by allowing the regions near the crack flanks to become unloaded.

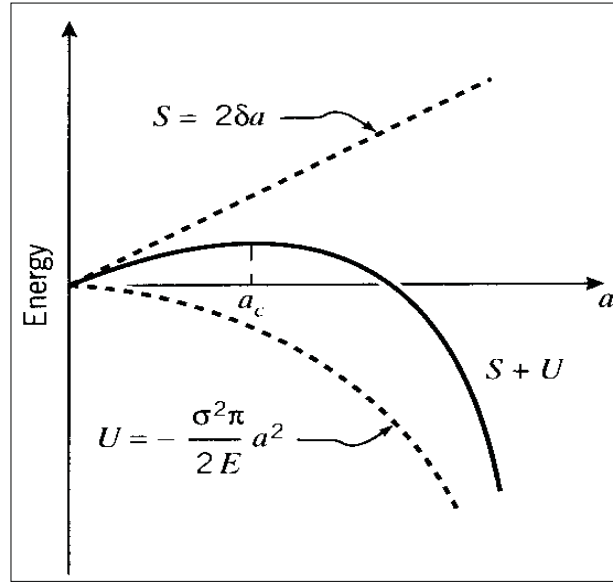


Figure 16. The fracture energy balance [16].

As the crack grows longer (an increase), the quadratic dependence of strain energy on a eventually dominates the surface energy, and beyond a critical crack length a_c the system can lower its energy by letting the crack grow still longer. Up to the point where $a = a_c$, the crack will grow only if the stress is increased. Beyond that point, crack growth is spontaneous and catastrophic.

The value of the critical crack length can be found by setting the derivative of the total energy $S + U$ to zero:

$$\frac{\partial(S+U)}{\partial a} = 2\gamma - \frac{\sigma_f^2}{E} \pi a = 0 \quad (5)$$

Since fast fracture is imminent when this condition is satisfied, we write the stress as σ_f . Solving,

$$\sigma_f = \sqrt{\frac{2E\gamma}{\pi a}} \quad (6)$$

Griffith's original work dealt with very brittle materials, specifically glass rods. When the material exhibits more ductility, consideration of the surface energy alone fails to provide an accurate model for fracture. This deficiency was later remedied, at least in part, independently by Irwin [16] and Orowan [17].

They suggested that in a ductile material a good deal – in fact the vast majority – of the released strain energy was absorbed not by creating new surfaces, but by energy dissipation due to plastic flow in the material near the crack tip. They suggested that catastrophic fracture occurs when the strain energy is released at a rate sufficient to satisfy the needs of all these energy “sinks,” and denoted this critical strain energy release rate by the parameter G_c ; the Griffith equation can then be rewritten in the form:

$$\sigma_f = \sqrt{\frac{EG_c}{\pi a}} \quad (7)$$

This expression describes, in a very succinct way, the interrelation between three important aspects of the fracture process: the material, as evidenced in the critical strain energy release rate G_c ; the stress level σ_f ; and the size, a , of the flaw. In a design situation, one might choose a value of a based on the smallest crack that could be easily detected. Then for a given material with its associated value of G_c , the safe level of stress σ_f could be determined. The structure would then be sized so as to keep the working stress comfortably below this critical value.

2.4 Experimental measurement of G

Used in everyday applications, adhesive joints are loaded in various modes. It is of major importance to determine the adhesive toughness for each mode in order to combine them with different mixity.

2.4.1 Mode I

The double cantilever beam (DCB) test is the most commonly used test method for mode I characterization. It follows ASTM D3433-99 standard, where a specimen (Figure 17) with an initial region not bonded with length a_0 is loaded in order to register the load P and displacement δ for each crack length a (Figure 18). Speed test must be defined in order to propagate the crack one minute after loading, resulting in values that range from 0.5 to 3 mm/min.

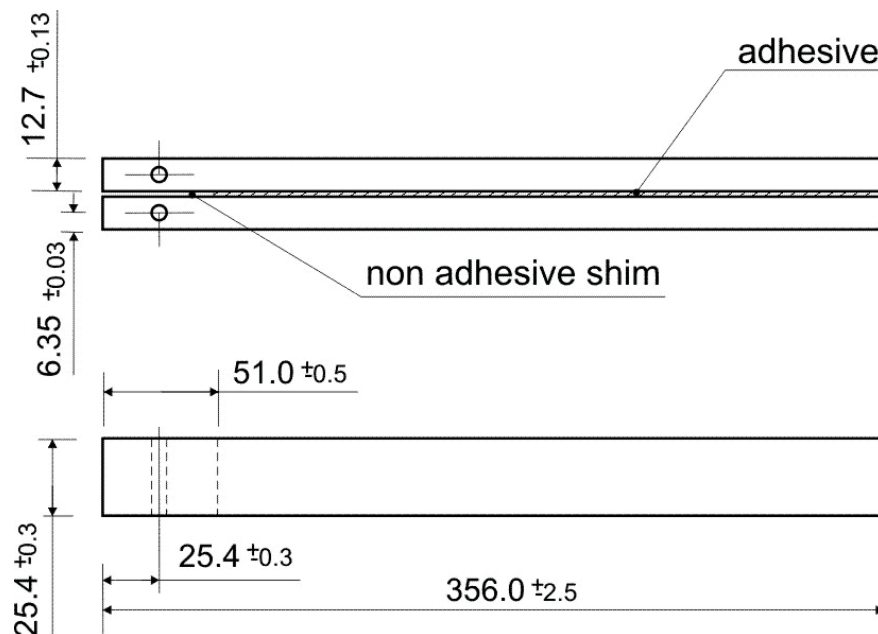


Figure 17. Double cantilever beam specimen geometry as defined in ASTM D3433 (dimension in mm) [18].

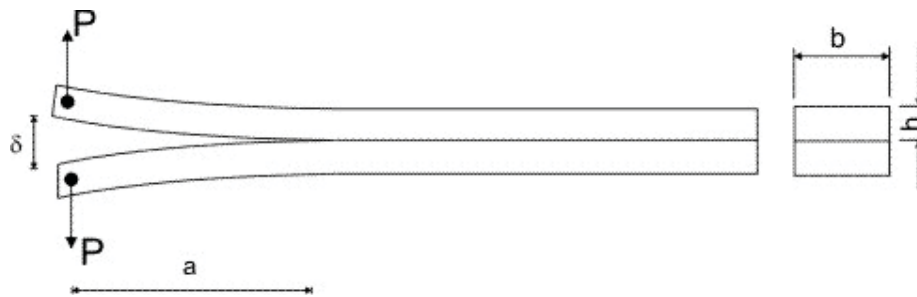


Figure 18. Schematic of DCB test [18].

ASTM D3433-99 standard defines another specimen named contoured double-cantilever beam (CDCB), also known as tapered double-cantilever beam (TDCB) allowing the measurement of the fracture toughness G_1 to be independent of the crack length a . Figure 19 shows its geometry.

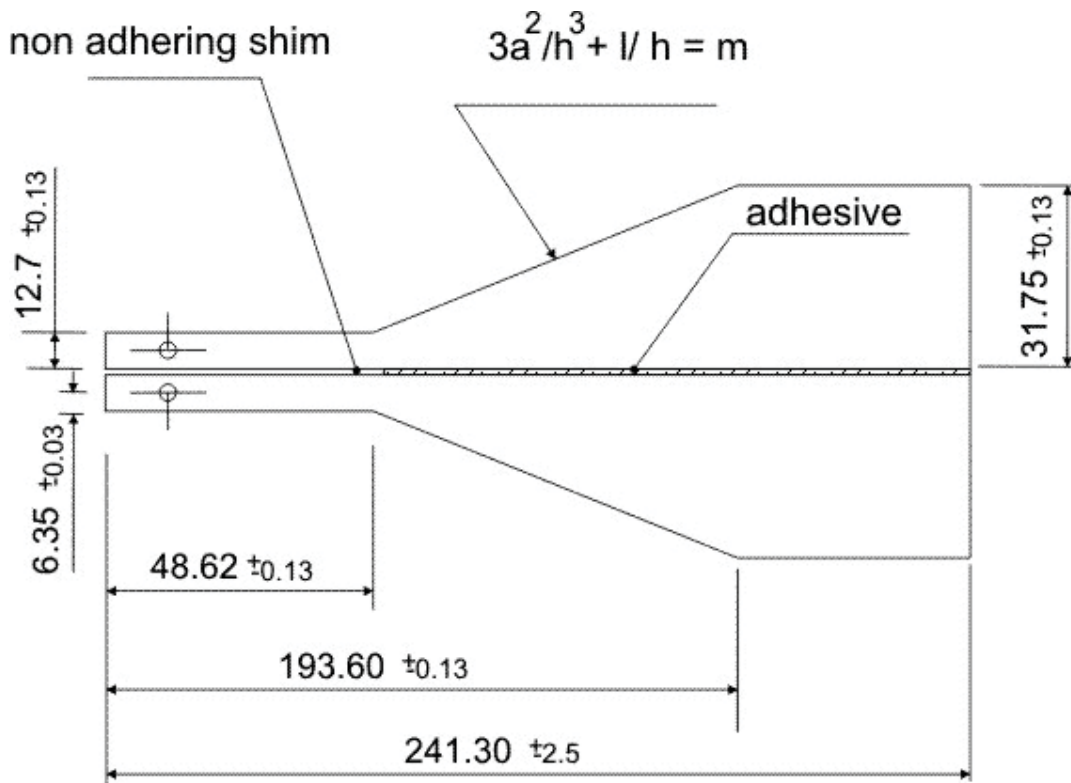


Figure 19. TDCB specimen [18].

Test method

The specimens are fixed to the testing machine with pins and then loaded at a crosshead separation rate chosen to keep time-to-fracture in the order of 1 minute. Load must be applied until point A is reached as shown in Figure 18 for DCB specimen and Figure 19 for TDCB. From this point forward, the crack begins to grow rapidly. Loading must be stopped and the crack growth curve in the chart must be followed until the load levels at an approximate constant value (the crack stops growing). At this point, the following values must be determined and recorded:

- Load to start crack – L max.;
- Load when crack stops – L min.;
- Distance from loading end of specimen to the stationary crack tip.

These records should be repeated five times for each specimen.

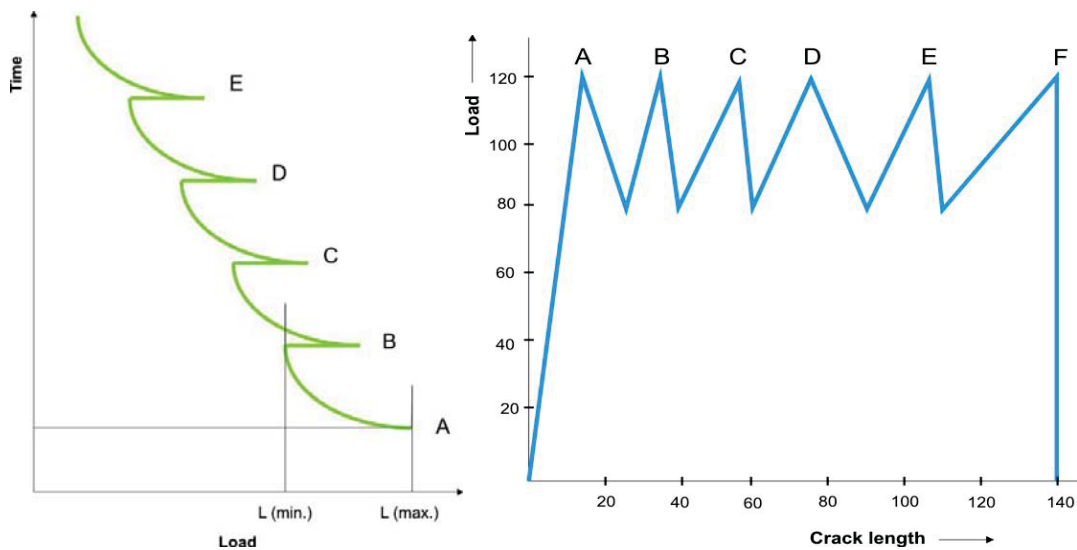


Figure 20. Typical test plots [18].

Closed form solution for G_{IC}

Assuming each adherent as a beam with the same length as the crack size (an in Figure 20), strain energy resulting from flexure and shear results in equation 9 [19].

$$U = 2 \left(\int_0^a \frac{M_f^2}{2EI} dx + \int_0^a \int_{-h/2}^{h/2} \frac{\tau^2}{2G} b dy dx \right) \quad (9)$$

where E is the Young's module, G is the shear module, and:

$$I = bh^3/12 \quad (10)$$

$$\tau = \frac{3P}{3A} \left(1 - \frac{y^2}{c^2} \right) \quad (11)$$

$$A = bh \quad (12)$$

$$c = h/2 \quad (13)$$

Using Castigliano's theory and solving equation 1 results in equation 6 that determines the displacement at the specimen end.

$$\delta = \frac{dU}{dP} = \frac{8Pa^2}{EBh^3} + \frac{12Pa}{5Gbh} \quad (14)$$

Accounting for flexibility $C = \delta/P$ and considering the Irvin-Kyes relation (equation 15), the critical energy release rate for the mode I is determined by equation 16:

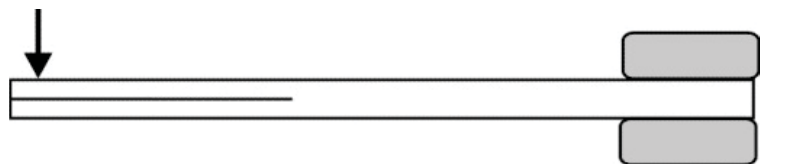
$$G_c = \frac{P^2}{2b} \frac{dC}{da} \quad (15)$$

$$G_{IC} = \frac{6P^2}{b^2 h^3} \left(\frac{2a^2}{E} + \frac{h^2}{5G} \right) \quad (16)$$

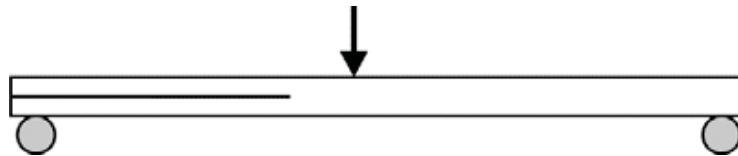
However this model does not account for the root rotation, i.e., the specimen rotation at the crack tip. Kannien [20] proposed a corrected beam theory considering a correction of the crack length determined by the compliance analysis.

2.4.2 Mode II

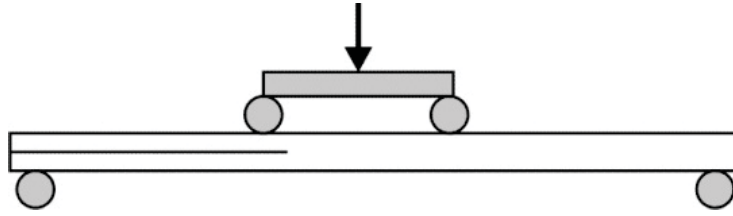
Mode II adhesive joint failure testing has no standard to define it. However, for composite materials testing, end notched flexure (ENF), end load split (ELS) and four-point end notched flexure showed in Figure 21 are used.



a) End loaded split



b) End notched flexure



c) Four-point end notched flexure

Figure 21. Mode II interlayer failures of composite materials tests [18].

The end loaded split test is sensitive to the gripping conditions and presents some troubles to determine G_{IIC} for large displacements [19]. The four-point end notched flexure requires a special apparatus and presents some difficulties with friction at the pre-crack region [20]. Despite the difficulty to measure the crack length, the end notched flexure test (ENF) is widely used, mostly because of its simplicity (see Figure 22).

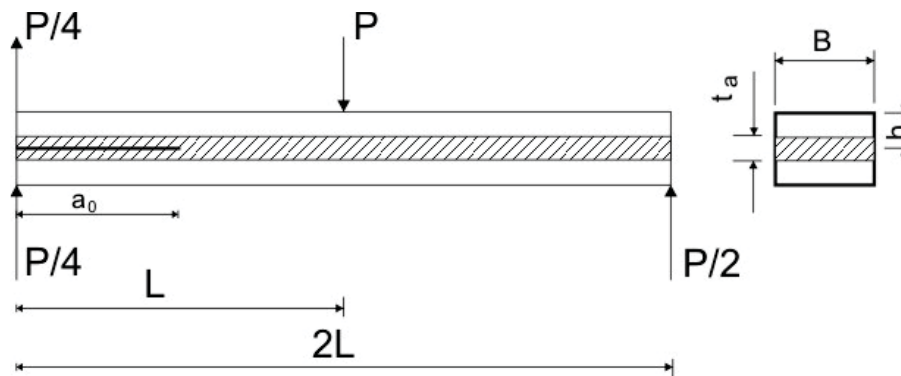


Figure 22. ENF test diagram [18].

Test method

The specimen is placed over two rods that will support its weight and the loading, allowing the rotation. The rods are placed symmetrical in relation to the loading point that should be half length of the specimen. During load history the

values of force, applied displacement and crack length (P , δ and a) are registered in order to calculate the critical strain energy release rate. Most of times it is difficult to accurately measure the crack length. To overcome this difficulty, de Moura et al. [21] proposed a new method, which does not rely on the crack length.

Closed Form Solution for G_{IIC}

Applying the beam theory analysis to each specimen adherent which supports half of the applied load (P), the strain energy is calculated according to equation 17.

$$U = \int_0^{2L} \frac{M_f^2}{2EI} dx + \int_0^{2L} \int_{-h}^h \frac{\tau^2}{2G} b dy dx \quad (17)$$

Analyzing the beam in three segments ($0 \leq x \leq a$; $a \leq x \leq L$; $L \leq x \leq 2L$), equation 17 becomes equation 18 for the flexure component and equation 3 for the shear component of the energy.

$$U_f = 2 \int_0^a \frac{(\frac{Px}{4})^2}{2EI} dx + \int_a^L \frac{(\frac{Px}{2})^2}{2EI} dx + \int_L^{2L} \frac{(\frac{Px}{2} - P(x-L))^2}{2EI} dx \quad (18)$$

$$U_s = 2 \int_0^a \int_{-h/2}^{h/2} \frac{\tau^2(c-\frac{h}{2})}{2G} b dy dx + \int_a^{2L} \int_{-h}^h \frac{\tau^2(c=h)}{2G} b dy dx \quad (19)$$

Using equation 19 for τ and applying Castiglione's theorem, the displacement of the load application point (shown in Figure 22) is given by equation 20 that differentiates into equation 21.

$$\delta = \frac{dU}{dP} = \frac{P(3a^3+2L^3)}{12Ei} + \frac{3PL}{10Gb h} \quad (20)$$

$$G_{IIc} = \frac{2P^2A^2}{16b^2Eh^3} \quad (21)$$

Besides the difficulty to measure the crack length, there is another drawback related with the existence of a region damaged by non-elastic processes, named fracture process zone (FPZ), illustrated in Figure 23. Silva et al. [22] found that the energy released at this fracture process zone must be accounted and an equivalent crack length that contemplates this effect must be defined.

$$G_{IIc} = \frac{9P_{II}^2 a_{eq}^2}{16B^2 E_{fII} h^3} \quad (22)$$

The presented methodology allows obtaining the strain energies G_I and G_{II} using only the P- δ data. For this reason it is named Compliance-Based Beam Method (CBBM).

Using this method it is not necessary to measure the crack length during propagation because the calculated equivalent crack length is used instead of the real one. Another advantage is related to the fact that a_{eq} includes the effect of the FPZ, which is not taken into account when the real crack length is considered.

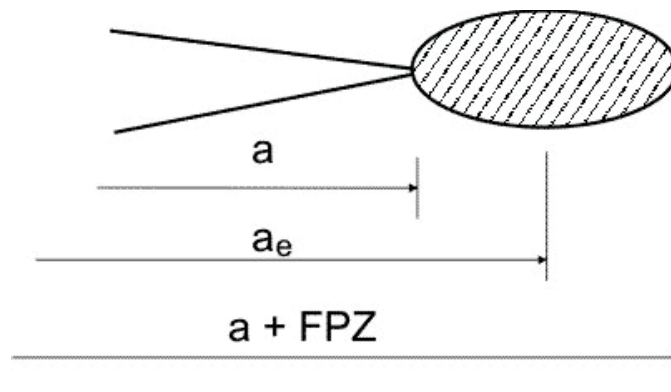


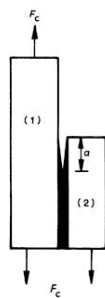
Figure 23. Schematic of the fracture process zone [18].

2.4.3 Mode mixity

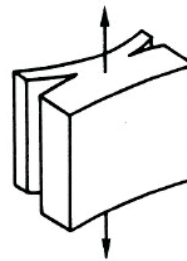
Generally, adhesive joints are loaded in a mixed-mode of shear and peel stresses resulting in a combination of mode I and mode II.

Mode mixity is important because the energy required to grow a crack along an interface is sensitive to the mode mixity of the stress field near the crack tip. It typically takes several times as much energy to grow a crack under mode II conditions than under mode I conditions. Anderson et al. [23,24] reported from their study with polyurethane/PMMA bonds that the adhesive fracture energies measured under different loading modes were ordered as mode I < mode II < mode III, coincidentally in ratios of approximately 1:2:3.

The development of experimental methods to determine the fracture properties for adhesives under mixed mode loading has been studied and implemented with some proposed specimens like CLS (cracked lap shear in Figure 24 a)), EDT (edge delamination tension in Figure 24 b)) [25], Arcan [26] used to vary the mode mixity (Figure 24c) , asymmetrical DCB [27] (Figure 24d)), mixed mode flexure test [28] (Figure 24e)), variable mixed mode test [29] (Figure 24f)) and mixed mode bending (MMB) [30,31].



a) Cracked lap shear specimen



b) Edge delamination tension specimen

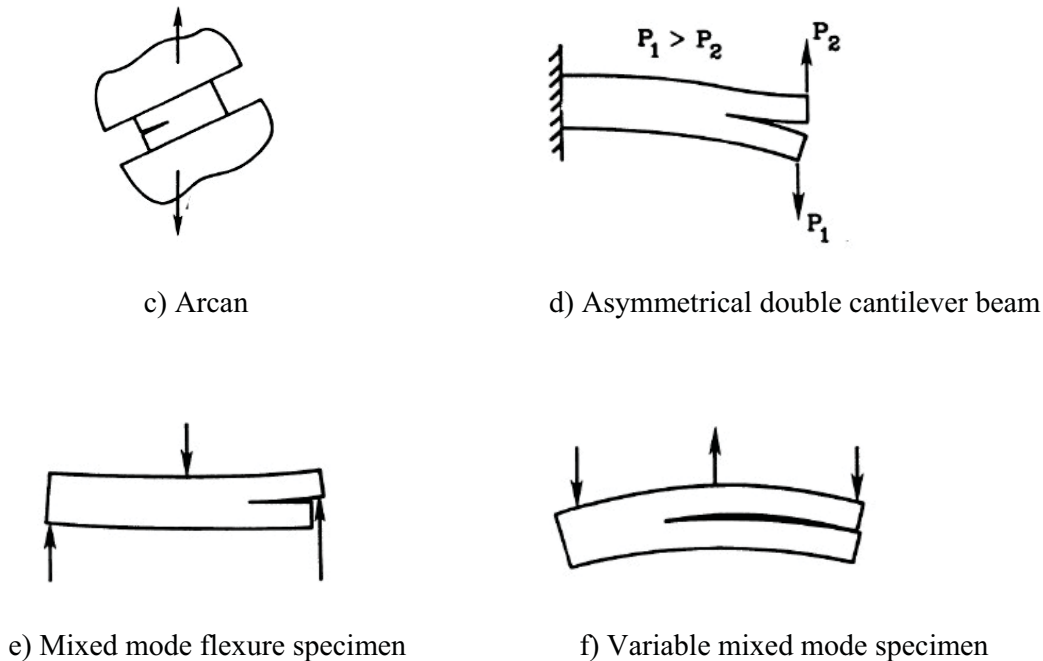


Figure 24. Mixed mode testing methods [18].

In comparison to all the other tests, the MMB configuration has:

- the possibility of using the same specimen geometry as for mode I tests;
- the means to vary simply the mixed mode ratio over the whole range from pure mode I to pure mode II.

As a result, this is the only specimen seriously being considered for standardization at present, used in ASTM Test Method D6671-01 for composites delamination testing.

The MMB test combines the mode I DCB test with the mode II ENF test. A special lever adds an opening-mode load to a midspan loaded ENF test, separating the arms of the specimen as in a DCB test. Changing the lever geometry (distance c in Figure 25) modifies the relative magnitude of the applied loads determining the mixed-mode ratio.

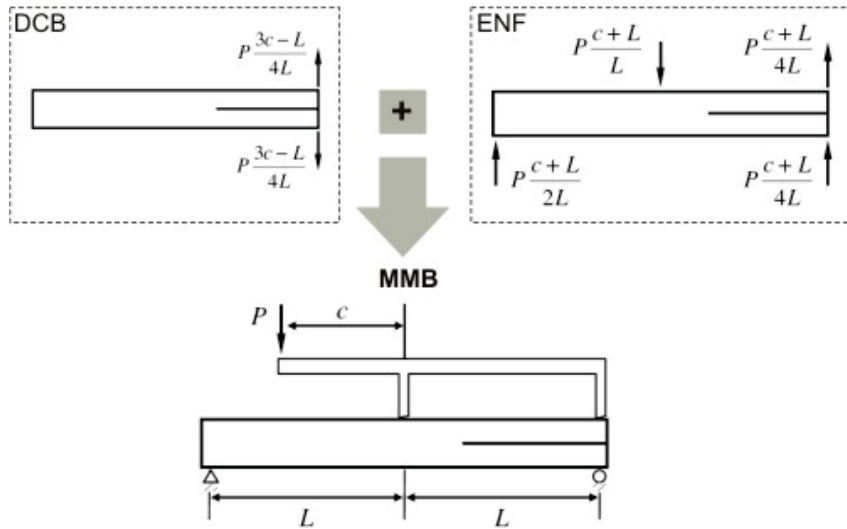


Figure 25. The MMB is a combination of DCB and ENF [18].

The beam theory can be used to analyze the mixed mode bending test.

Test method

Mixed mode testing, as defined in ASTM D6671, requires an apparatus to split laminate specimens to determine the delamination or debonding fracture toughness at various mode ratios. The base of the apparatus holds the specimen while the lever loads it through hinges, end tabs or clevis pins. The lever roller acts as a fulcrum, pushing down the lever arm opposite the tab or pins that are pulled up. Mixed mode ratio is controlled by the length of the lever arm. The load must remain vertical during the loading process. To prevent the geometric nonlinear effects due to lever rotation, the height of loading is slightly above the pivot point where the lever attaches to the test specimen.

The fracture toughness, G_C , and mode mixture, G_C/G are calculated from critical loads registered in a load versus displacement curve. This load displacement curve should be recorded on a x-y recorder, equivalent real-time plotting device or stored digitally.

Closed Form Solution for G_{Ic+IIc}

Mixed mode bending can be represented by a superposition of DCB and ENF, and is governed by the following equations [23, 24]:

$$G_I = \frac{12a^2 P_I^2}{B^2 h^3 E_L} \quad (23)$$

$$G_{II} = \frac{9a^2 P_{II}^2}{16B^2 h^3 E_L} \quad (24)$$

and so

$$\frac{G_I}{G_{II}} = \frac{4}{3} \left[\frac{3c-L}{c+L} \right]^2 \quad (25)$$

where:

$$P_I = \left(\frac{3c-L}{4L} \right) P \quad (26)$$

$$P_{II} = \left(\frac{c+L}{L} \right) P \quad (27)$$

2.5 Influence of the adhesive layer thickness

The thickness of the adhesive layer contributes for the joint behavior. Thus it should be taken into account and thoroughly studied. Boscom et al [33, 34] found that fracture energy is maximized when the adhesive layer thickness equals the diameter of the fracture process zone (FPZ) ahead of the crack tip.

Kinloch and Shaw [35] showed that the FPZ played an important role in enhancing GIC of the adhesive joint. Daghyani et al. [36, 37] found a transition in the fracture process from a cohesive to an interfacial adhesive failure for thin layers. Lee et al. [38] found that the thickness effect could be explained by the different coefficients of thermal expansion between the adherend and the adhesive. The same work showed that as bond thickness decreases, the fracture energy either decreases monotonically (type A), or increases, peaks, and then decreases rapidly (type B) as shown in Figure 26.

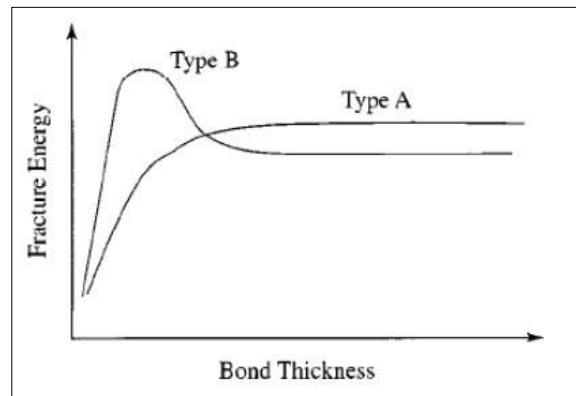


Figure 26. Two typical types of bond-thickness effects on the fracture toughness of adhesive joints [38].

The graph in Figure 26 should be studied to understand which type of adhesives are Type A and Type B, and what magnitudes of fracture energy and bond thickness are at stake.

In adhesive bonding it is important to understand that the adhesive layer applied in between the two bonded bodies is usually very thin (of the order 0.05 to 0.2 mm), thus it behaves differently compared to the adhesive as a bulk material. Gleich et al. [39] determined that the maximum values for G_c are obtained when the adhesive thickness is nearly the diameter of the plasticized area.

If it is true that thicker adhesive layers result in bad joint properties, when the adhesive layer becomes thinner than the surface roughness it is difficult to promote the connection between the two surfaces because it is difficult to fill the voids.

The ability to absorb energy, characterizing ductile or fragile adhesive plays also an important role when evaluating the bondline thickness effect. Figure 27 a) and b) shows that the fracture process zone is affecting the adherends and the interface, while Figure 27 c) the same fracture process zone does not affect the adherends or the interface.

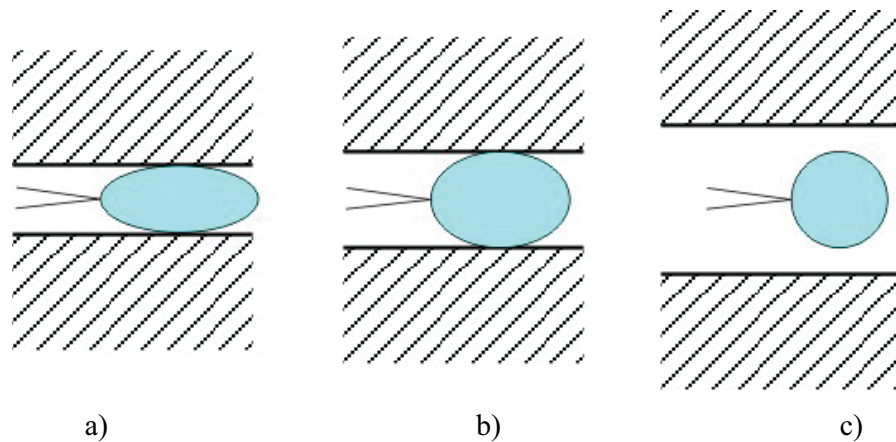


Figure 27. Bond line thickness effect affecting the fracture process zone (thickness $a < b < c$) [39].

Thickness has a greater impact when using ductile adhesives in comparison to brittle ones. Kinloch [35] states that the thickness of the adhesive layer usually does not affect the measured value of the adhesive fracture energy or brittle adhesives, as for the tougher ones this parameter may significantly affect the measured value of the fracture energy. This is explained by the plasticity that occurs in the vicinity of the crack tip.

TESTING PROCEDURES

3.1 Introduction

This study is about the determination of the toughness in mode II for different types of adhesives. The ENF test was used for its simplicity and because it is well documented.

The geometry considered was the same as that for the DCB, to prove that same geometry can be used for the both type of tests. Initially, the adhesive thickness was supposed to be varied but due to time limitation only 1mm thickness was studied.

3.2 Specimen Manufacture

A good specimen is fundamental to achieve cohesive failure and obtain the best test data. Therefore, manufacture of specimens must be very well attended. The specimens manufacture should be done according to the following steps:

1. Mould Preparation;
2. Substrate preparation;
3. Adhesive mixture (when needed) and deposition;
4. Hydraulic press;
5. Curing of the adhesive;
6. Removal and cleaning;
7. Preparation for testing.

3.2.1 Mould Preparation

The mould in Figure 28 was used to produce the bonded joints. Six specimens at a time can be made with this mould.

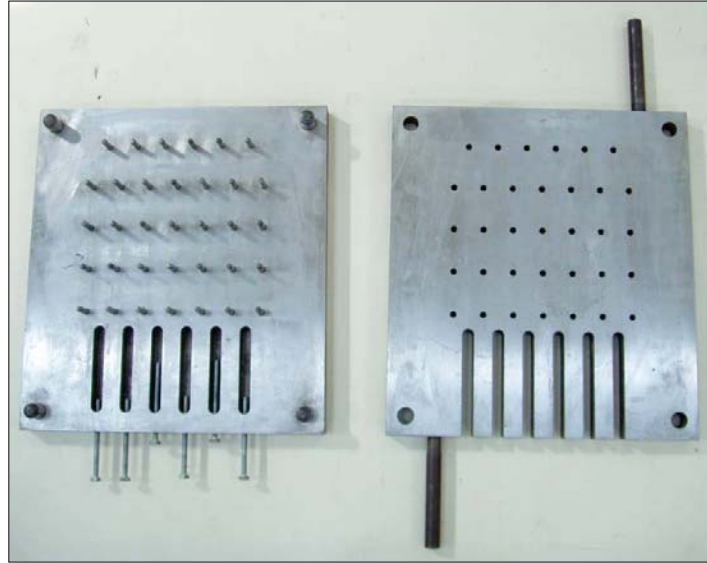


Figure 28. Mould used to prepare de bonded joints.

Before any use of the mould we have to guarantee a perfect clean surface without any burs and remains portions of adhesive. If that happens we must remove it with an aluminum spatula (Figure 29) and then pass acetone using a clean cloth (Figure 30).



Figure 29. Remove remains of adhesive.



Figure 30. Cleaning with acetone and paper sheet.

After cleaning the mould, we covered the surface with a release agent. In this case the FREKOTE 770NC.

The parts were heated to 60°C in a hot press to speed up the cure reaction of the release agent. After the parts reached 45 °C, three coats were applied with 5 minutes interval between. The time was the necessary to let the release agent cure. All parts must be coated carefully. All security procedures were used to avoid health damage. The razor blade and the spacers were surface-treated with release agent before the specimen parts assembly.



Figure 31. Mould release agent application

3.2.2 Substrate preparation-Sand blasting

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

Beams must be placed inside the sandblast machine and the surface must be sandblasted.



Figure 32. Specimens placement inside the sandblasting machine



Figure 33. Working with the sandblaster

Figure 33 shows the operator with his hands in the sandblaster protective gloves, looking through the screen and pressing the foot actuator for the jet airflow.

The surface should be sandblasted in a manner that the final finishing looks like sand, with high rugosity.

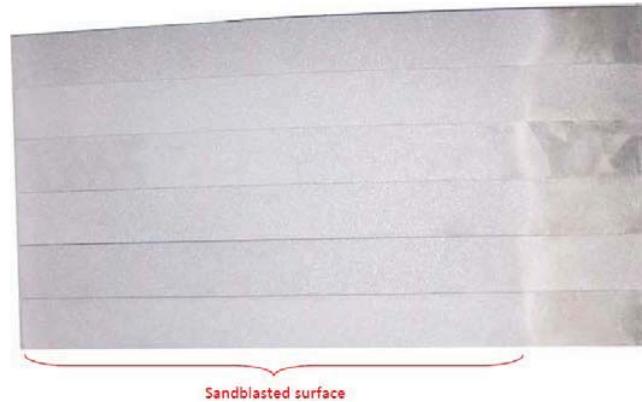


Figure 34. Sandblasted surface in the beams.

3.2.3 Adhesive preparation and deposition

To avoid problems with the adhesive pot life, the bottom beam must be already placed in the mould and prepared with the spacers and razorblades before the adhesive is applied.

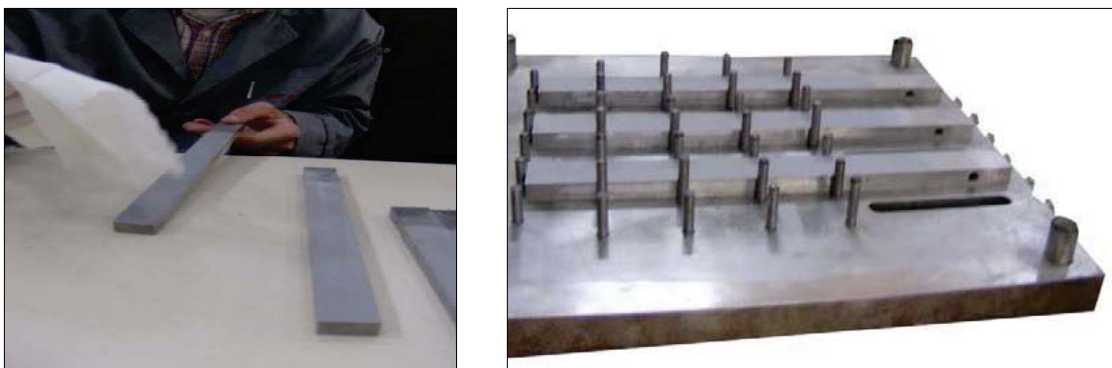


Figure 35. Cleaning of the substrate with a paper tissue (left) and placing the bottom substrate in the mould (right).



Figure 36. Application of the top spacers (gauge feeler in the left and razorblade assembly in the right).

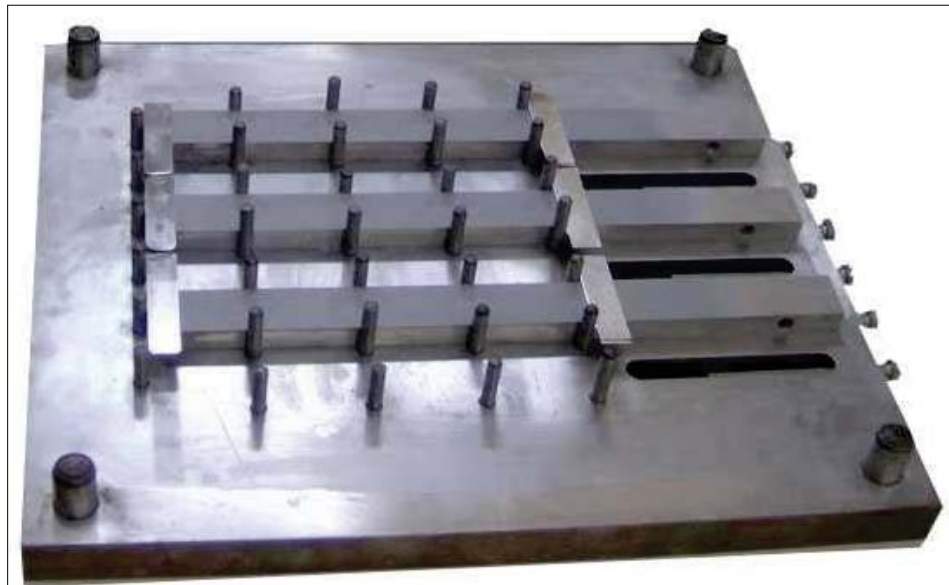


Figure 37. Three bottom substrate prepared with spacers and razorblades assemblies.

The next step is to apply the adhesive. The majority of structural adhesives is obtained by mixing a resin with a hardener that works like a catalizer. Some adhesives are supplied in dual container cartridges that are attached to a deposition mixing nozzle (Figure 38) which promotes the mixture of the two components, avoiding the operator to mix them manually. Both Araldite 2015 and SIKAFLEX 255 have mixing nozzles.



Figure 38. Mixing nozzle.

Araldite AV138M is supplied in a separate container from the hardener HV 998, as show in Figure 38 a.



Figure 39 – a) Adhesive AV138M and hardener separate containers and b) mixture tools to mix the two components.

These two components need to be mixed manually in a proportion of 100 parts of AV138M to 40 parts of Hardener HV998, as show in Figure 38 b.

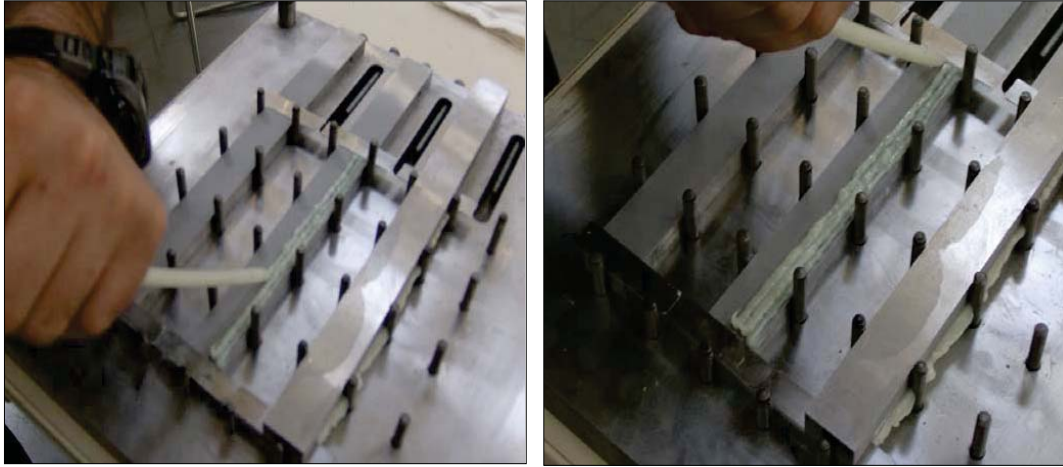


Figure 40.Araldite 2015 application.

The Araldite 2015 is applied in strings substrate (Figure 40). After the application of the adhesive the upper substrate of the specimen must be placed against the bottom substrate (Figure 41).

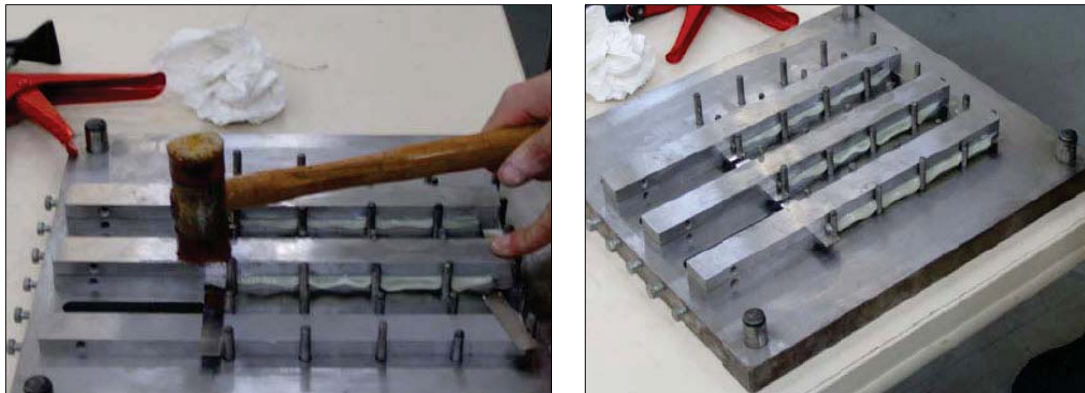


Figure 41.Application of top substrate.

For SIKAFLEX 255 FX, there is the need to apply previously a 206 G+P primer. The primer (206 G+P) must be applied just as a paint with a brush (Figure 42).

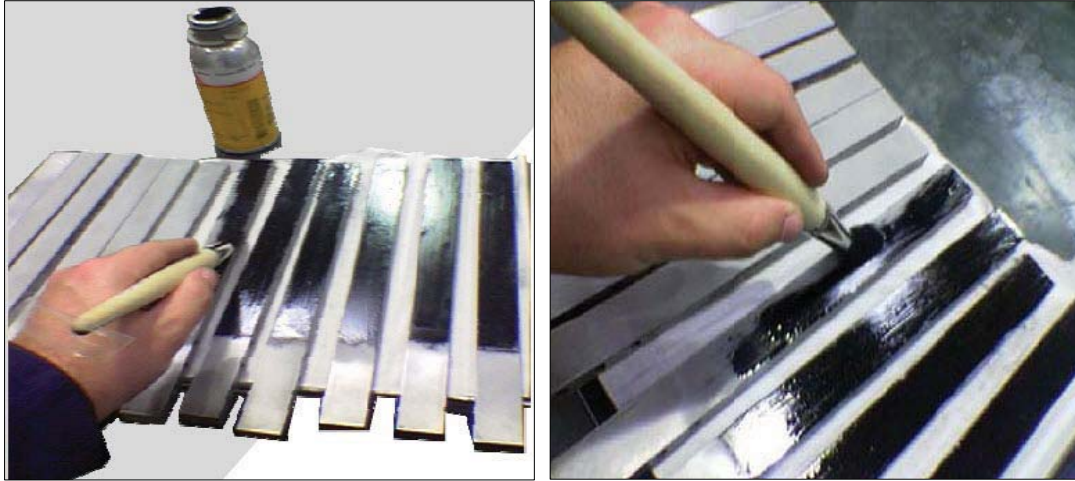


Figure 42. Applying the primer for SIKAFLEX 255 – FX.

3.2.4 Hydraulic press

When all the top beams are placed, the mould top plate must also be placed (Figure 43).



Figure 43. Mould closed

The assembly of the mould closed and the specimens must be taken into the hydraulic heated press from Intoco and a pressure must be applied by closing the pressure valve until the load cell reading gets to 1000 kg.

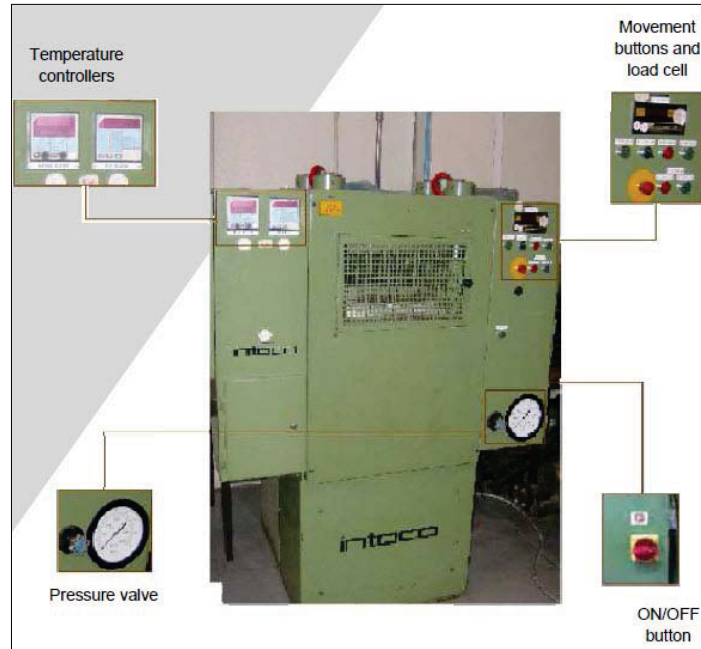


Figure 44. Intoco heated plate's hydraulic press machine.

There alignment of the specimens in the mould should be verified before applying the full pressure.

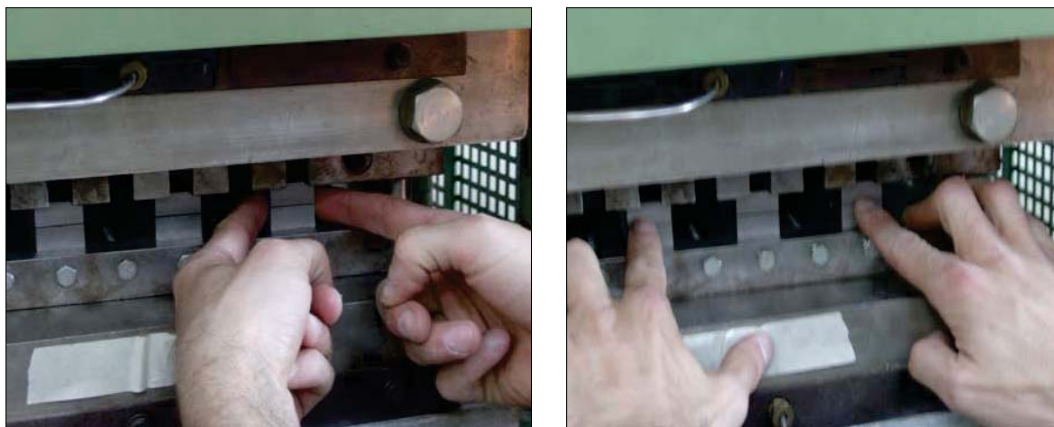


Figure 45. Verifying the lateral (left) and top (right) alignments.

3.2.5 Curing of the adhesive

The temperature must be set in accordance with the adhesive curing time specifications, defined by the manufacturer. The next table summarizes the temperature and curing time for each adhesive.

Temperatures and curing times.

Adhesive	Temperature [°C]	Curing Time	
		in the press	outside the press
SIKAFLEX 255 FX	Ambient (20)	24 Hours	one week
AV138 + HV998	80	30 minutes	two days
Araldite 2015	60	45 minutes	two days

3.2.6 Removal and cleaning

After the curing time in the press, the mould should be removed and the specimens can be taken out the mold. There may be the need for removing the pins of the mold. There is a proper tool from BETA and a hammer for that operation.

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.

3.2.7 Preparation for testing

Once the specimen is free of the adhesive in excess, the glue line must be clearly visible and maintain a constant thickness. A coat of white paint was applied over the glue line. A corrector painter is usually the best option for that.

After being painted in white, side rulers or scales can be attached or glued, helping the measurement of the crack length while it is growing during the test.

3.3 Test method

Geometry of specimen

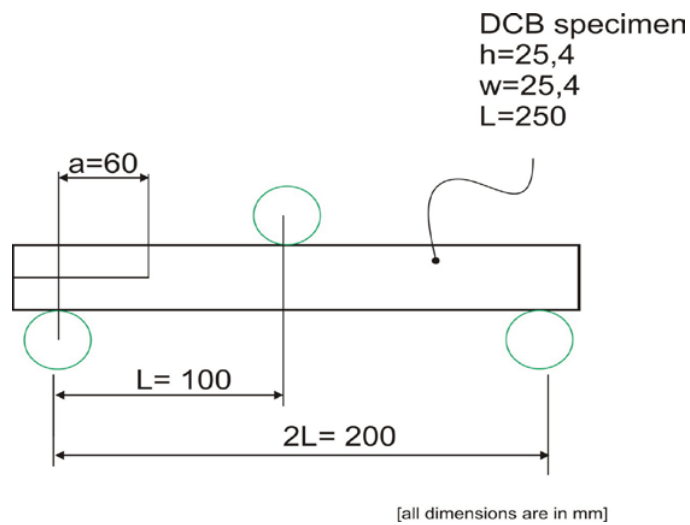


Figure 46.ENF Specimen geometry parameters.

The fracture toughness can be calculated equation 28.

$$G_{IIC} = \frac{9a^2 P^2}{16w^2 h^3 E_1} \left[1 + 0.2 \left(\frac{h}{a} \right)^2 \frac{E_1}{G_{13}} \right] \quad (28)$$

where,

a – crack length;

h- specimen height;

w- specimen width;

E_1 - adherend (steel) E1;

G_{13} - adherend (steel) G13.

ENF test

The specimen is placed over two rods that will support its weight and the loading, allowing the rotation. The rods are placed symmetrical in relation to the loading point that should be half length of the specimen, as shown in Figure 47.

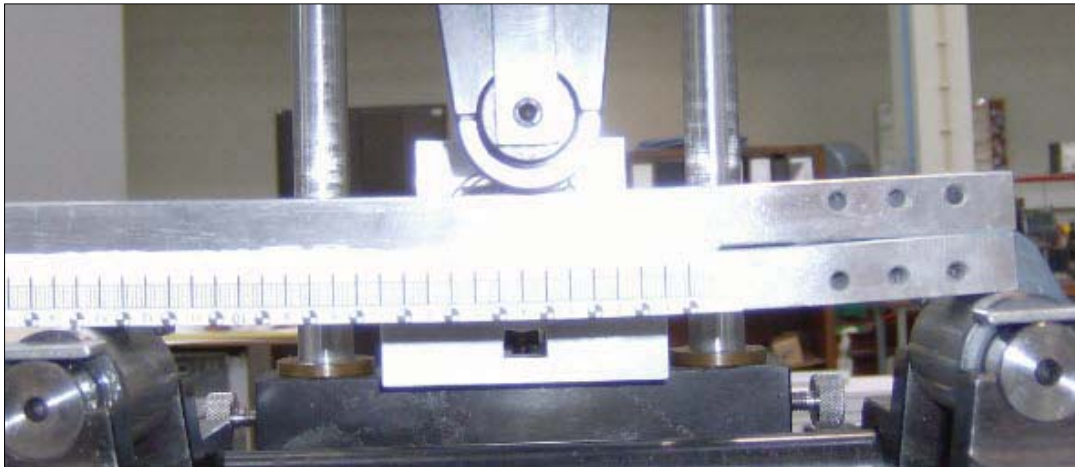


Figure 47. Test specimen

During load history the values of force, applied displacement and crack length (P , δ and a) are registered in order to calculate the critical strain energy release rate. Most of times it is difficult to accurately measure the crack length. To solve this problem, a high speed camera was used to record crack length.

Tests were conducted at room temperature at a constant displacement rate of 1 mm/min using the MTS 312.31 servo-hydraulic machine (Figure 48). Loads and displacements to failure were recorded with high speed camera. Nine joints were tested.

During the testing of specimens, to record the development process the following equipment was used: a high speed camera, a computer that has incorporated a data acquisition boards and software that is installed in computer.



Figure 48.MTS machine.

RESULTS AND DISCUSSION

4.1 Adhesive AV138

The fracture surface obtained for this adhesive is show in Figure 49.

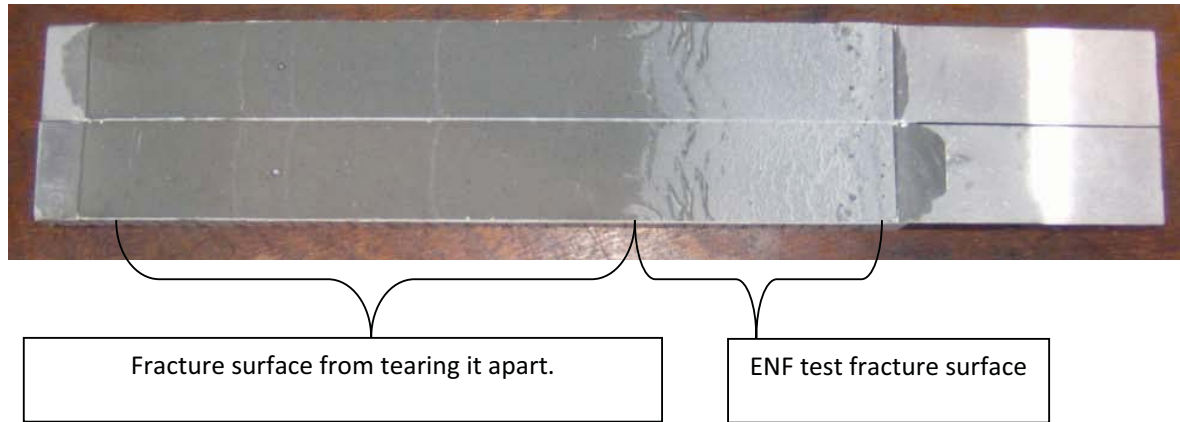


Figure 49. Fracture surface in ENF test for AV138

Only the right part corresponds to mode II crack propagation. When the crack reached the loading pin, the test was stopped. In effect from that point the loading is mixed due to the mode I compression of the loading pin.

The load-displacement curve for the AV138 is showed in next figure.

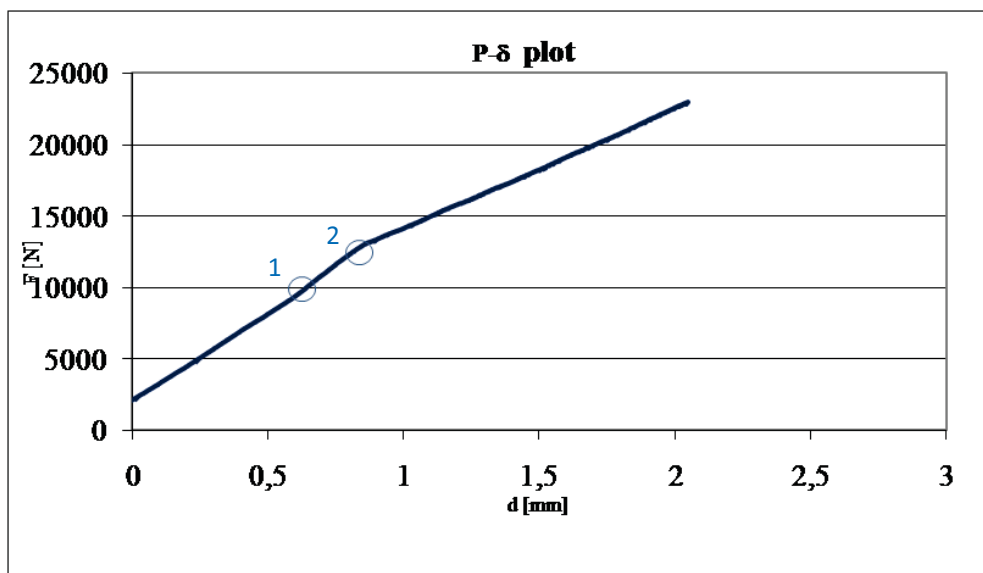


Figure 50. P-δ plot for the AV138 ENF test.

Maximum force to reach crack growth initiation is close to 15000 N.

The P- δ curve shows 3 distinct zones. From point 1(see Figure 50) the stiffness increases because the two substrates are sliding at the end of specimens. From point 2, the crack propagates. The crack propagation was instable quickly reached the loading pin.

That is why the toughness in the R-curve (Figure 51) has only a very narrow band corresponding to mode II crack propagation.

In order to obtain more stable crack propagation a longer length between the crack tip and the loading point should be used.

The graph for R-curve is illustrated in Figure 51. The value of G_{IIc} were obtained with de method of de Moura et al [21].

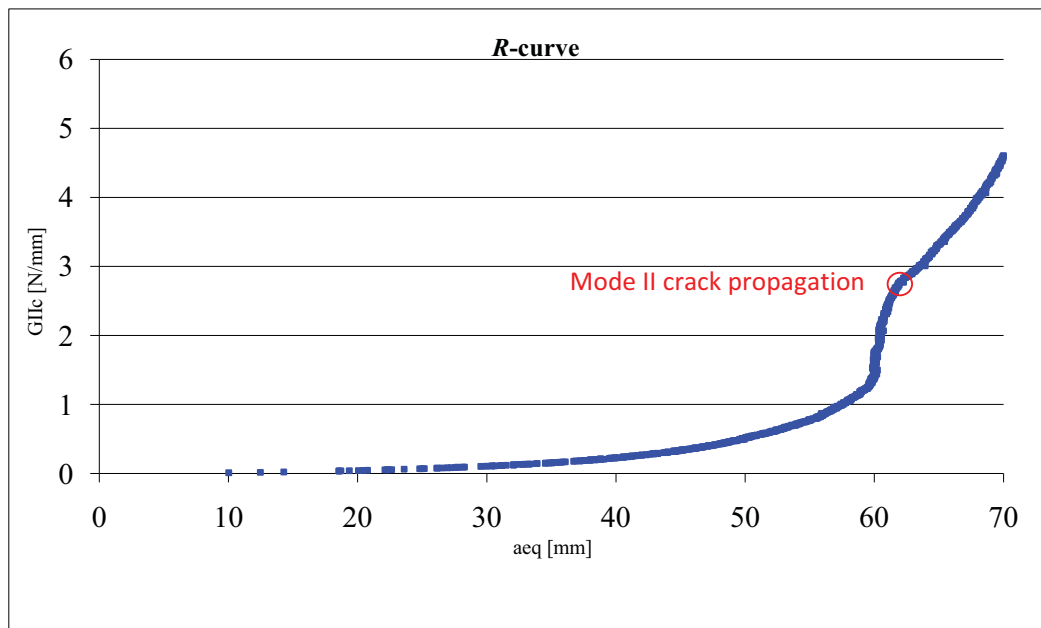


Figure 51.R- curve for the AV138 ENF test

The value of G_{IIc} given by the narrow band in Figure 51 was approximately 2,7 N/mm .The value of G_{Ic} for this adhesive is 0,35 N/mm [40].That makes a relation of $\frac{G_{IIc}}{G_{Ic}}$ of approximately 15.

4.2 Adhesive Araldite 2015

The fracture surface obtained is shown in Figure 52.

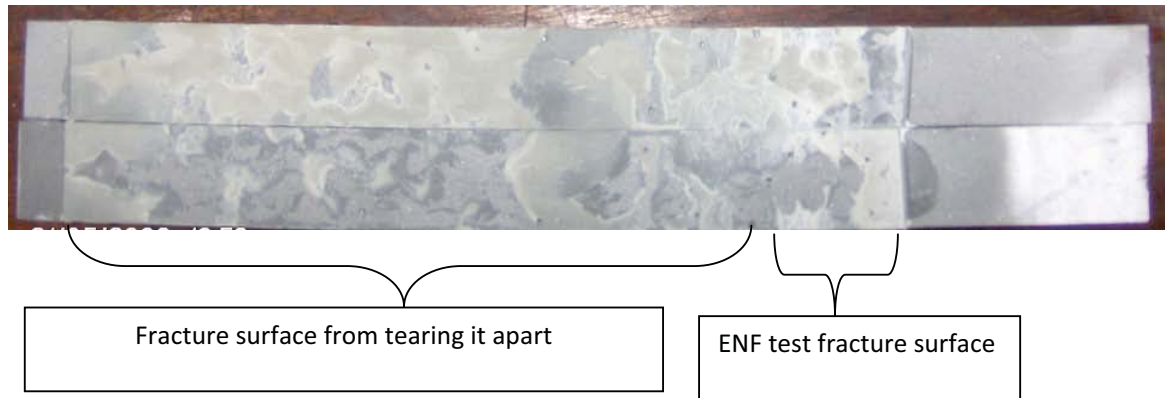


Figure 52. Fracture surface in ENF test for Araldite 2015

The P- δ curve for the Araldite 2015 is shown in Figure 53.

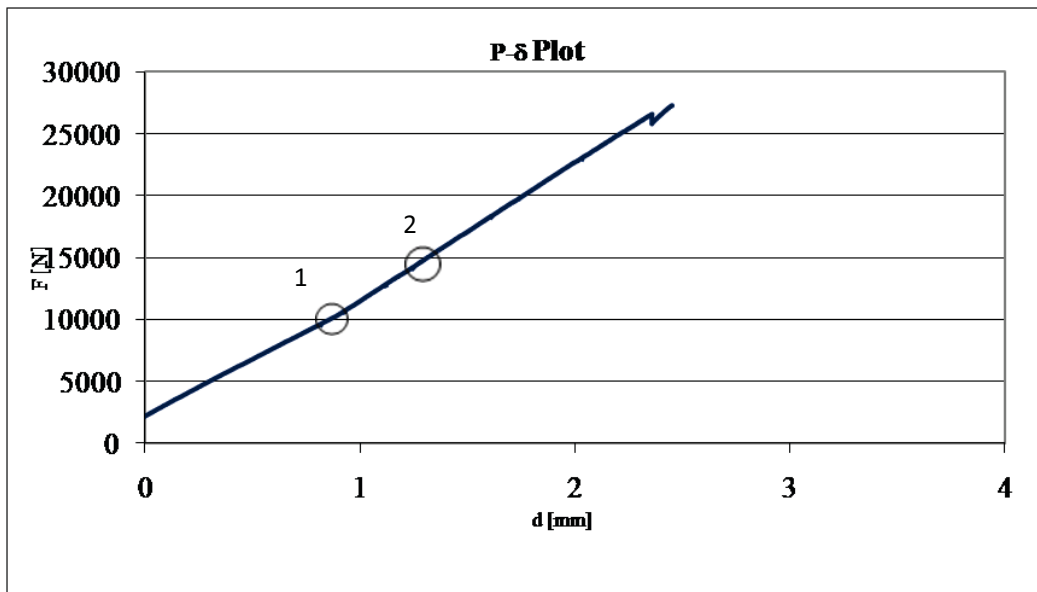


Figure 53. P- δ curve for the Araldite 2015 ENF test

The P- δ curve for Araldite 2015 does not show distinctively the three zones observed for the AV138 adhesive. But still, point 1 and 2 of Figure 53 can be defined.

Also, the narrow band in the R-curve is not distinguishable. Nevertheless, a value for G_{IIC} was extracted based in point 2 of Figure 53.

R-Curve for the Araldite 2015 ENF test is illustrated in Figure 54.

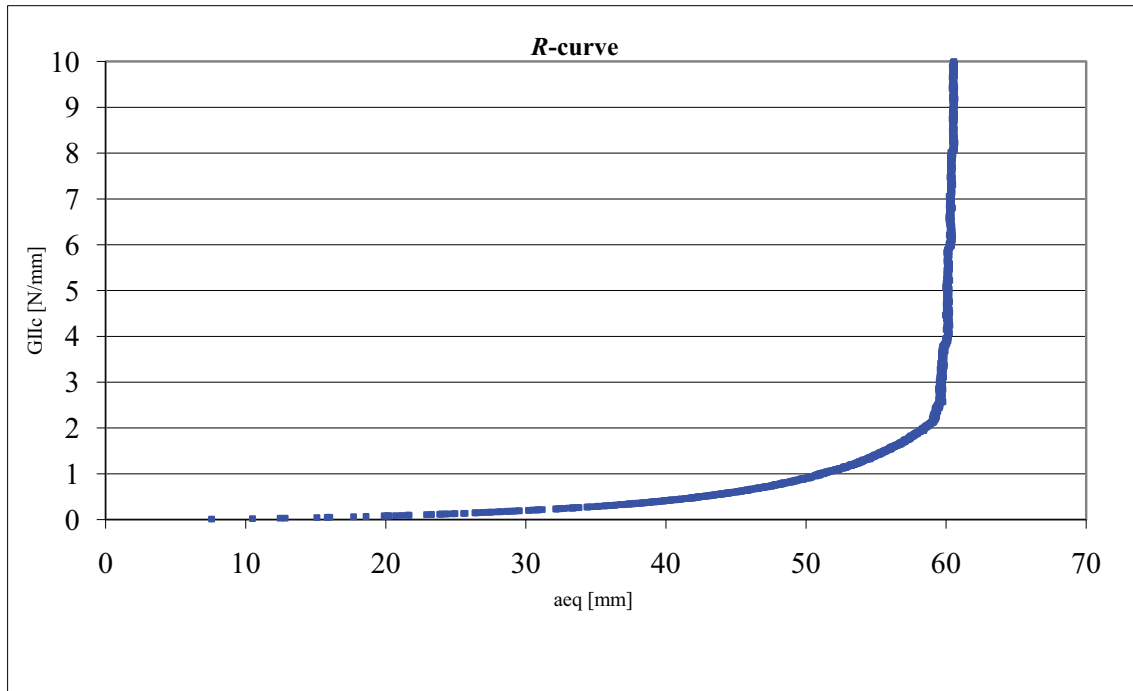


Figure 54.R-curve for the Araldite 2015 ENF test

After 60 mm, there is no inflection point at the curve, so this was not a valid test. Nevertheless, we know that the crack occurred at 14200 N, so can say from the data gathered that there should be 4,24 N/mm for the energy release rate G_{IIc} .

Using this point, a value of $G_{IIc}=4,24$ N/mm was obtained. Again, these values represent more the 10 times $G_{IC}[40]$ and proves that the toughness in shear is much higher than in peel.

The specimens with SIKAFLEX 255FX was manufactured but there was no time to test specimens.

Chapter five

CONCLUSIONS

The most important results are that is possible to use only one geometry for both mode I and mode II test and, the adhesive toughness in shear is approximately ten times the toughness in peel.

Mode II test using the ENF loading and the DCB geometry were conducted for one brittle adhesive (AV138) and ductile adhesive (2015)

The results are promising for the brittle adhesive AV138 with an energy release rate value of 2,7 N/mm. For the Araldite 2015, the crack growth was unstable and its R-Curve did not present an inflection point preventing to obtain a good value for the fracture release energy. This may be corrected by changing the pre-crack length allowing to obtain a bigger path for the crack before it reaches the loading cylinder. It is also likely to obtain stable crack propagation by reducing the adhesive layer thickness.

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