Chapter 2
Adhesive Bonding

2.1 Introduction

A thorough understanding of the types of defects that frequently occur in adhesively bonded joints is required. Any defect found in an adhesive bond has the potential to affect the strength of that bond. Industry requires a method of not only identifying such defects but also the setting of threshold criteria, to define acceptable/unacceptable defects. The threshold criteria are dependent on where the joint is to be deployed and tailored its application. The first step in this process is to categorise types of defects in adhesive bonds and then to develop reliable methods of NDE that can identify the defect type before tailoring the threshold criteria.

There are two fundamental methods in which an adhesive adheres to a substrate, mechanical interlocking and chemical interaction. Mechanical interlocking is dependent on the adherend surface finish. If a surface is very smooth there is no opportunity for mechanical interlocking, whereas if a surface is too rough air may become trapped in the troughs of the surface [1]. Both situations will reduce bond strength as the area of actual bonding is decreased. If any contamination, such as dust, is present on the adherend surface the adhesive will adhere to it, instead of the adherend, again reducing the total bonded area and weakening the joint. Chemical interactions or chemical bonding is governed by the Van der Waals forces interacting between molecules [2]. The combined strength of these forces varies for different types of materials and affects the surface energy (SE) of the adherend [3]. For a good bond to be produced a high SE is preferable, as this attracts the adhesive and enhances surface wetting [4]. A very low SE repels other materials and so bonding is much more difficult. Typically plastics tend to have low SE and so generally present challenges during bonding [2]. Polypropylene has the lowest surface energy of thermoplastics at 31.2 mN m^{-1}, epoxy resin has a surface energy of 32.9 mN m^{-1}, both very low when compared to metals and glass which have a surface energies of 1000–5000 and 300 mN m^{-1} respectively [5]. Most issues associated with bonding can be mitigated by appropriate adherend surface

R.C. Waugh, *Development of Infrared Techniques for Practical Defect Identification in Bonded Joints*, Springer Theses,
DOI 10.1007/978-3-319-22982-9_2

preparation and adhesive selection. Unfortunately in large structures perfect surface preparation is difficult to achieve and there is a risk of contamination so there is a requirement to accurately inspect if defects have formed during the bonding process.

2.2 Defects in Adhesive Bonds

Several types of defects occur in adhesive bonds [6, 7]. These may be considered to fall into three main categories, see Fig. 2.1. The first type is an inclusion, which is the physical inclusion of a foreign material in the adhesive joint, as in Fig. 2.1a. This could occur, for example, if positioning tape is left on the adherend and has adhesive applied over it. The second type are voids, which is the inclusion of air in the joint, see Fig. 2.1b. This could occur between the adhesive and adherend during joint assembly or be found in the adhesive itself if air is introduced during mixing of a two part adhesive. Voids may also be found in the adherend. The final category of defect are kissing defects, see Fig. 2.1c. Kissing defects are the result of improper adhesion between the adhesive and the adherend where the adhesive/adherend interface is not as strong as expected for that joint configuration [8]. A decreased level of adhesion leads to reduced joint strength and is a significant threat to structural integrity. As all components of the joint are present and in contact, kissing defects are the most difficult type of defect to detect using NDE techniques as very little material property contrast is created.

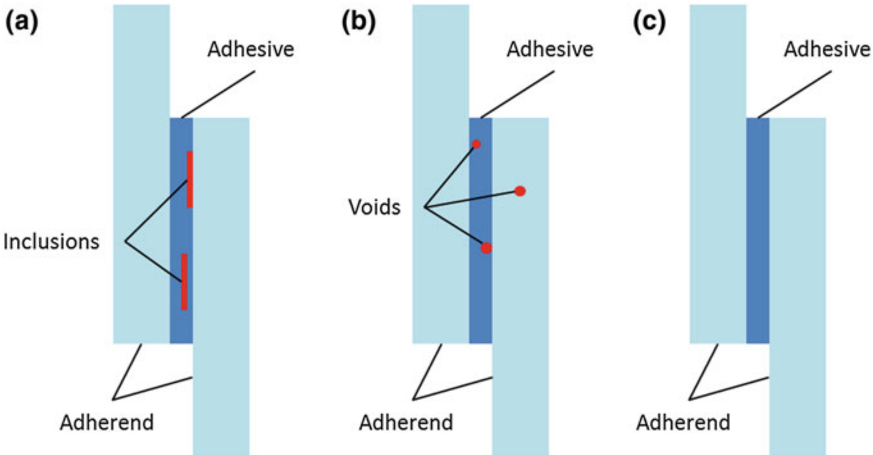

Fig. 2.1 Three categories of defects typically found in adhesive joints; **a** inclusions, **b** voids and **c** kissing defects

2.2.1 *Adhesive Bond Failure*

Adhesive bonds may fail in three ways [9]:

- Adherend failure
- Cohesive failure
- Adhesive failure

If the bond fails via adherend failure the bond has been properly designed and manufactured, as the bond is not the weakest part of the structure and the adherend material has been used to its full potential. This type of failure is not due to problems with the bond. Cohesive failure is failure within the adhesive itself. This may be caused by excessive shear or peel stresses in the bond resulting from a poor joint design and is characterised by the presence of adhesive on both adherend surfaces of a failed joint. Adhesive failure is the failure between the adhesive and adherend, also known as interfacial failure and is caused by the improper preparation or pairing of the adherend and adhesive pairing. Adhesive failure is reported to only occur if there is a problem in the initial joint construction and is not caused through fatigue of the joint [10].

It is noted that for the early failure to be attributed to a kissing defect the failure mode must be adhesive, i.e. at the interface between the adhesive and adherend. As an attempt to quantitatively define a kissing defect a minimum strength reduction of 80 % of the shear strength of a perfect joint found during a lap shear test has been suggested to confirm that joints contained kissing defects [11]. It is acknowledged that this criteria is partly arbitrary, although is based on the observed nature of kissing defects.

2.3 Laboratory Created Defects

In order to develop and understand NDE techniques components or specimens containing defects must be used. There are several methods commonly used for the creation of defects in a laboratory in metallic and composite samples. The most common methods shall be discussed in this section as well as a discussion specifically concerning the simulation of kissing defects.

Flat bottom holes

One of the most common techniques used to simulate defects in metallic materials and ceramics is to mill a flat bottom hole in the rear of the sample thus creating a locally thinned material to simulate a defect [12]. This method originated as a method of simulating corrosion in metals but has been used for other purposes and applied to the study of composites. While this method was appropriate to simulate the loss of material associated with corrosion it is not a realistic means of simulating a commonly occurring defect in composite materials that are not prone to corrosion although due to the strong contrast that flat bottom holes create they are often used when developing techniques in initial studies [13].

Inserts
The most common means of creating an experimentally simulated defect in a fibre reinforced composite material such as carbon fibre reinforced plastic (CFRP) and glass fibre reinforced plastic (GFRP) is the addition of an insert between plies during manufacture. There has been broad use of the addition of PTFE or Teflon inserts typically of circular or square geometry placed at various depths throughout the thickness of the component, e.g. [14–16]. PTFE inserts are typically used to simulate a delamination between plies. This type of experimentally simulated defect is easy to produce and gives an accurately known defect location and geometry.

'Real' damage
Impact damage is frequently introduced in a controlled manner with known impact energies to introduce a controlled experimentally simulated defect that is more realistic of a defect a component may acquire while in service, e.g. [17, 18]. Core crushing has also been introduced when looking at studying sandwich structures [15]. Finally, using composites it is generally possible to manufacture a sample that has an inherent weakness in its layup which is prone to failure in a particular mode and a particular location when subjected to loading. Such contrived samples have been used to produce delaminations in composites or disbonds in adhesive bonds [19]. The nature of creating a 'real' defect is that the precise location and geometry of the defect is not known prior to experiments therefore they can be used to conclusively parameterise defect comparisons of a developing technique. The technique must then be compared to an established NDE technique or destructive testing undertaken to verify results.

2.3.1 *Experimentally Simulated Kissing Defects*

Voids and inclusions are of known origin and are therefore relatively easy to recreate in the laboratory, however, the exact cause of kissing defects is unknown. Possible causes are thought to be contamination, abnormality in the adhesive chemistry or curing process, moisture ingress, residual stresses, or a combination of these factors [11, 20]. Several studies have focused on the recreation of kissing defects in the laboratory, e.g. [21]. Most have categorised kissing defects into two types, dry contact and liquid layer [22]. In a dry contact recreation, adhesive is applied to one adherend and cured. The other adherend is put in position and held in place by compressive loading, as shown in Fig. 2.2a. No actual adhesion occurs between the adhesive and adherend. A liquid layer defect is achieved by the introduction of a thin layer of a contaminant such as grease at the adhesive/adherend interface, Fig. 2.2b. The thickness of the contaminant is much thinner than the thickness of the adhesive layer but this thin defect has a detrimental effect on the strength of the joint.

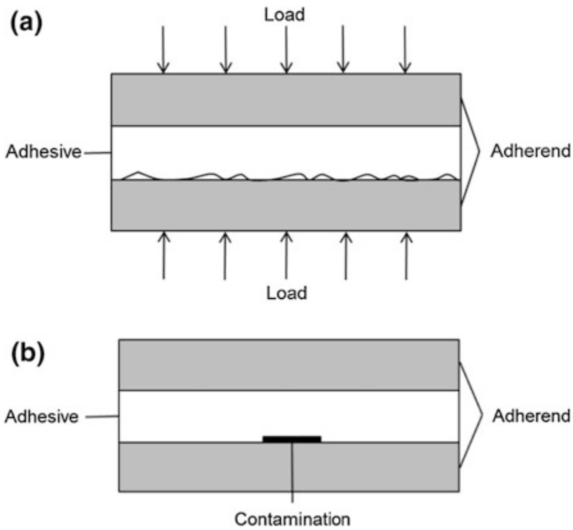

Fig. 2.2 Laboratory created defects, **a** a dry contact compressively loaded defect and **b** a liquid layer defect with a thin layer of contamination between adhesive and adherend

2.4 GTT MkIII Adhesive Bonding and Inspection Techniques

An application of concern in this work is the adhesively bonded Triplex joints found in the secondary membrane of GTT MkIII LNG carriers. As these joints are enclosed during construction of the vessel and unable to be accessed once completed, it is of paramount importance that they are inspected and found to be defect free, to avoid costly and disruptive remedial repair work having to be carried out during service.

2.4.1 Construction of Triplex Adhesive Bonds

In the MkIII carriers the liquefied natural gas (LNG) is stored in several tanks; each made using a membrane system. Working inwards from the inner hull of the ship, the system comprises of a secondary layer of insulation that is attached to the inner hull using mastic, a secondary membrane, a primary layer of insulation and a primary membrane which contains the LNG, see Fig. 2.3. The primary membrane is 1.2 mm thick stainless steel, which is corrugated to allow for thermal expansion. The 1 m × 3 m steel sheets are robotically welded together using a TIG (tungsten inert gas) process. The primary and secondary insulation blocks are typically 100 and 170 mm thick respectively and are both made of polyurethane foam surrounded by plywood. The secondary membrane is constructed using a specialised Triplex material, which is a 0.7 mm thick sheet of aluminium with a layer of glass cloth on either side attached using a resin system, the specifics of which are confidential.

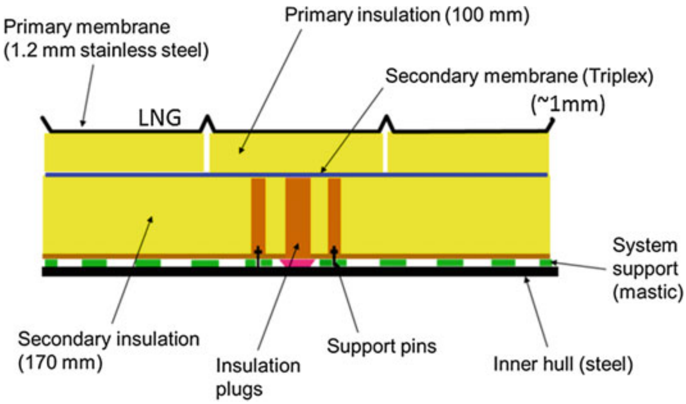

Fig. 2.3 Schematic diagram of the insulation system for the GTT MkIII type LNG carrier membrane system tanks [23]

Prefabricated units comprising both layers of insulation and the secondary membrane measuring 3 m × 1 m are brought into the shipyard where they are attached to the inner hull. A piece of flexible Triplex (FT) is then used to join the rigid Triplex (RT) on the prefabricated boxes to form a continuous secondary membrane, Fig. 2.4. The only difference between the two Triplex materials is the type of resin used to bond the glass fibre to the aluminium, which determines its flexible or rigid nature. In the RT epoxy resin is used and infused into the glass fibre whereas a layer of rubber adhesive is added in the FT. This difference allows the FT to be flexible, enabling easier application. Currently the FT is attached to the RT using either epoxy adhesive, which is applied by hand, or polyurethane (PU) adhesive, which is usually applied by an automated process but only in very

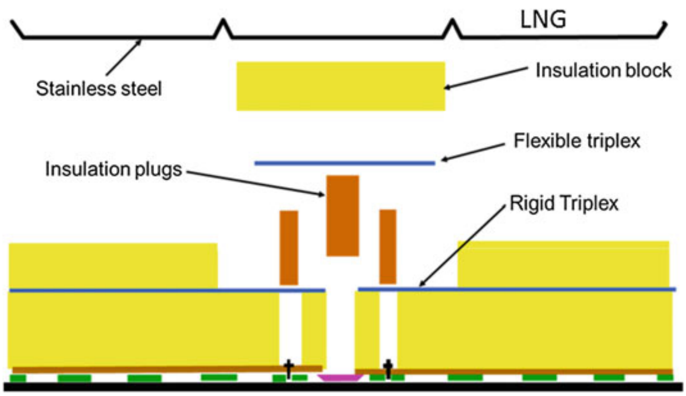

Fig. 2.4 Construction of adhesive Triplex bonds in the secondary membrane of the GTT MkIII insulation system [23]

small, specific areas of the ship. Due to the improved properties of the PU adhesive at low temperatures compared to the epoxy, there is a push towards using an increased amount of the PU. Epoxy remains the most widely used as manipulation times for the PU are insufficient for the manual application procedures used in most areas of the ships.

The vast majority of prefabricated units delivered to the shipyard are 3 m × 1 m, with special pieces being fabricated for the corners and more complex areas. These blocks are adhesively bonded and pinned to the inner hull [24]. Any gap between the prefabricated blocks is filled with more insulation. Adhesive is then applied to the RT and the FT is overlaid. The adhesive is hand spread to a thickness of between 0.2–0.8 mm, with an ideal thickness of 0.4 mm. In the epoxy adhesive a thickness of greater than 0.8 mm becomes too brittle when exposed to low temperatures and when the adhesive is less than 0.2 mm thick the strength of bonding is reduced. Although reducing the range of acceptable adhesive thicknesses would improve the quality of the joint while a manual application procedure is employed it is impractical to do so. The amount of adhesive applied is monitored by using a known weight of adhesive for a set area, which is typically a 3 m long bond or 0.3 m width. A pressure bag is then used to apply 150 g/cm^2 of pressure, although pressures in the range 120–400 g/cm^2 are acceptable. Pressures above 400 g/cm^2 are not accepted as this leads to the adhesive being squeezed out from the joint and pressures less than 120 g/cm^2 are considered insufficient to adhere the Triplex fully. The pressure is applied for 12 h as the epoxy cures.

Tank spaces found in the MkIII are 60 m × 33 m × 75 m and in the current fleets there are 5 of these tanks. The total length of adhesive joints to be assessed in a single MkIII carrier is currently 50–60 km. There are plans to increase the size of the carriers to accommodate 12 tanks in the future, more than doubling the length of adhesive joints that require inspection.

2.4.2 Current Testing Techniques for Triplex Bonds

Once the bond has cured and the pressure bag has been removed a series of inspection techniques are carried out. Squeezed out adhesive along the edges of the bonds is visually inspected. The quality of this squeezed out adhesive has been found to be indicative of the quality of the adhesive in the joint. For example, if there are air bubbles in the squeezed out adhesive there are typically air bubbles in the joint, leading to a reduction in the quality of the joint. In another visual test the surface of the FT is illuminated to allow shadows to be cast by any bumps on the surface. These could indicate any defects caused by larger air bubbles or any solid contamination. Another manual test is to take a thin object, such as a coin, and perform a tap test across the surface. Defects that cause a discontinuity below the surface of the material can be heard by the inspector. The effectiveness of this method is dependent on the skill and experience of the inspector. A final test that is carried out on junctions between different pieces of FT is a local tightness test [25].

A vacuum is applied encompassing the full width of the FT and sealed onto the RT. A soap solution is applied to the surface of the joint and as the vacuum is applied the joints are monitored to see if any bubbles form. These bubbles indicate that air is being pulled through the joint from the insulation side by the vacuum and so the joint is not air tight and may allow the LNG to leak through to the inner hull.

Once the construction is complete a total tightness test is carried out [25]. Leaks are detected by sensors between the primary and secondary membranes and the secondary membrane and the inner hull during construction. Nitrogen gas is pumped into the gap between the primary and secondary membrane. The detectors in the space between the secondary membrane and the inner hull monitor for leaks. Whilst this method allows leaks to be detected, it is extremely difficult to locate them accurately and expensive to repair them once they are already sealed into the membrane system.

If a defect or leak is detected by using any of the previously mentioned inspection techniques the joint must be removed and replaced or repaired. This is a costly process due to the nature and size of the joints. For example, if a leak is detected during the total tightness test then the primary membrane and primary insulation must be removed to access the joint. The Triplex joint must then be disassembled without causing damage to the rigid Triplex or secondary insulation and the joint must be remanufactured. The remaining layers must then be reattached and retested. This is a very time consuming process that should be avoided if at all possible. If a leak is found during the vessel's service then the ship must be taken out of service, costing the ship owners a substantial amount of money in revenue as well as the cost of the repair, which currently totals in the region of $15 million (USD) per incident depending on the extent of the repair required (D. Howarth, Lloyds Register, personal communication 30th March 2010).

As has been described, most current inspection techniques are manual. The practicalities of this lead to only selected 'high risk' areas of the ships being routinely tested, as a full inspection is impractical. Therefore there is demand for an automated inspection method that could quickly collect and process the data, identifying any problem bonds. The total length of the bonds of interest dictates that a time efficient NDE approach must be developed in order for it to be feasible for application in a shipyard.

2.4.3 Current Research on Triplex Bonds

The main subject of research related to the membrane style LNG carriers is sloshing investigations, e.g. [26, 27]. These studies focus on the effect of sloshing impact of the LNG on the membrane system and mitigation of excessive transfer of impact load to the primary insulation layer via the introduction of composite mats between the primary membrane and the primary insulation. The structural reliability of the membrane containment system under various capacity conditions has been investigated. This research has led to increased awareness of the most dangerous capacities to carry and so has resulted in new loading guidelines being established [28]. Other real scale experimental tests have been carried out focusing on the structural behaviour of the secondary barrier in a MkIII LNG carrier [29]. The structural response was studied for thermal and mechanical loading. Strains were measured via strain gauges and fibre Bragg gratings in the adhesive layer. This experimental work was then correlated with a finite element model. Three adhesive systems were compared: the standard epoxy adhesive used, the PU adhesive previously mentioned and an epoxy adhesive cured using a heat pad. Whilst it was found that the latter two greatly improve the structural properties of the joint the impracticalities associated with the short work time of the PU and the added cost of the heated cure epoxy meant that currently joints continue to be constructed using the standard epoxy. As the standard epoxy provides a safety factor of more than six [29], a change in adhesive is not necessary to withstand the structural loading imparted on the Triplex joints in service.

A further study has investigated the use of a vibration isolation system to reduce the impact of cavitation caused during LNG boil off transmitted between the primary membrane and the primary insulation [30]. An E-glass/epoxy composite corrugated plate type vibration isolation system to be located beneath the primary membrane was developed, which was found to successfully reduce impact and protect the primary foam insulation with good fatigue resistance.

Research has also been carried out focusing on a slightly varied membrane design where the secondary barrier is simply made of aluminium and the insulation used is polyurethane foam (PUF) [31]. Cracks originating in the aluminium and propagating through the foam at low temperatures were found to be an issue in this construction. The addition of glass fibre in the PUF was found to be a solution. As the aluminium found in the Triplex material is faced with glass fibre the secondary membrane found in the MkIII has not been reported to experience cracking problems.

The main issue reported with the secondary membrane of the MkIII carriers is the detection of leaks, which leads to extremely costly repairs being required. Kim et al. [32] have found the primary leakage path through the Triplex barrier to be at the interface between the flexible Triplex and the adhesive. It was shown that this was due to a lack of impregnation of the adhesive into the glass fabric caused by the high viscosity of the epoxy. Adapted curing cycles have been suggested, involving heating of the epoxy for an initial period, while fibre wetting takes place and then a reduced temperature while solidification of the epoxy occurs, thus avoiding introduction of thermal residual stresses. Whilst it was shown that this increased the adhesive impregnation of the fibres and thus reduced leakages it did not mention the cost associated with the heated curing regime and how realistic this is to implement in shipyards. Despite the promising results, to date there has been no application of this method and as such it can only be assumed that the cost associated with this is too great for ship builders to employ a heated curing cycle along the length of the joints in the MkIII carriers.

The International Code for the Construction and Equipment of Ships carrying Liquefied Natural Gas in Bulk [33] stipulates that the secondary membrane must be periodically checked ‘using an appropriate method’. Maguire [33] focuses on the use of global and local acoustic emission techniques to identify leaks through the membrane. To do this, sensors are positioned throughout the region of interest. The insulation space between the secondary membrane and the hull is taken to a partial vacuum, while the insulation between primary and secondary barriers remains at ambient pressure. Any leaks should then be detected by the sensors and their position may then be triangulated using additional sensors. Whilst this process is able to detect leaks, due to the scale of the vessels determining their precise location is relatively difficult and so can be a time consuming process. The application of this technique is also limited to quiet times on the ship as it is very sensitive to external noise. This technique is aimed at through life service monitoring, not for use during construction. For Triplex a good bond is believed to be a good bond for life and as such there is still a great demand for a reliable inspection technique for use during construction after the manufacturing of the Triplex joints and before the primary insulation is overlaid.

To date there has been no published work to the author’s knowledge on the application of an advanced NDE technique during construction that is able to assess the integrity of the secondary barrier in the Triplex material. The development of such a technique holds the potential to reduce costly repairs later in the construction stages and through life.

A NDE technique is to be developed that is suitable to provide inspection of the bonded joint between the FT and the RT in the secondary membrane of the MkIII LNG carrier. The inspection should take place during the construction of the vessel after the curing of the bond and before the joints are covered with primary insulation. Due to the length of the joints of interest an efficient, preferably automated, inspection technique is required. This work is concerned with the development of a suitable NDE technique and the on-site automation of the procedure will be a further stage of development.

2.5 Summary

The criteria of identifying kissing defects have been identified. A kissing defect must have an effect on the strength of the bonded joint and must be of approximately zero volume. If a bond is to fail due to the presence of a kissing defect then it must fail via adhesive failure. A review of methods of experimentally simulated defects has been presented, identifying several methods that have been applied to composite materials and adhesive bonds. A problem identifying kissing defects in the MkIII LNG carrier at the bondline between the primary and secondary insulation has been highlighted and it is known that current inspection procedures are unable to identify such defects. Current NDE techniques used across a wider range of industries shall be studied in the following chapter.

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http://www.springer.com/978-3-319-22981-2

Development of Infrared Techniques for Practical
Defect Identification in Bonded Joints
Waugh, R.C.
2016, XXVIII, 149 p., Hardcover
ISBN: 978-3-319-22981-2