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EVALUATION OF SOLUTIONS FOR ADHESIVE JOINING OF PEEK TO POLYMER ELECTROLYTE MEMBRANE

Dissertação no âmbito do Mestrado Integrado em Engenharia Mecânica na especialidade de Produção e Projeto orientada pela Professora Doutora Dulce Maria Esteves Rodrigues e pelo Professor Doutor Pedro Miguel dos Santos Vilaça da Silva e apresentada ao Departamento de Engenharia Mecânica da Faculdade de Ciências e Tecnologia da Universidade de Coimbra.

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EVALUATION OF SOLUTIONS FOR ADHESIVE JOINING OF PEEK TO POLYMER ELECTROLYTE MEMBRANE

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Avaliação de soluções para ligação por adesivo de PEEK com membrana polimérica eletrolítica

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To my family.

La science, mon garçon, est faite d'erreurs, mais d'erreurs qu'il est bon de commettre, car elles mènent peu à peu à la vérité.

Jules Verne, in *Voyage au centre de la Terre*, 1867.

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Abstract

To fight climate change and pave the way for a carbon-neutral economy, the European Union and its members have been implementing strategies that recognize the potential of hydrogen to support the global effort to reach the goals of the Paris Agreement. In the scope of this, innovation and improvement for systems that use hydrogen as supply, fuel or energy carrier is needed.

Polymer Electrolyte Membranes (PEM) fuel cells are a type of fuel cell that converts the chemical energy of hydrogen into electricity. Although these devices are promising for the transportation industry, they present manufacturing and reliability challenges.

Currently, the fabrication of fuel cells, which complies fuel cell stacks assembly, involves tightly pressing together the components in order to guarantee proper sealing. The fasteners used to keep the system in place, are not the most effective solution to insure sealing, and, at the same time, also show corrosion problems with usage. To tackle this issue, an alternative design, which discards the use of fasteners and demanding compression systems, during fuel cells assembly, is envisaged.

This work proposes the use of a polymer structural frame, to support the Polymer Electrolyte Membrane. This frame replaces the gasketed contact between the electrolyte and the bipolar plates, typical of traditional fuel cell designs. The structural polymer to be used in the fabrication of the structural frame is the polyetheretherketone (PEEK). Since the Polymer Electrolyte Membranes are made of Nafion, developing solutions for the dissimilar joining of the two polymers is of uttermost importance for the development of the new fuel cell design.

Based on the above-described problem, the experimental work performed under the scope of this investigation was planned with the objective of solving the challenge associated with the dissimilar joining of the PEEK and PEM polymers by adhesive bonding. Since the two polymers have low surface energies, its joining by adhesive bonding is an engineering challenge, which comprises not only the selection of a proper adhesive for the envisaged application, but also developing a suitable joint design.

The work performed involved the selection of a suitable adhesive among a cyanoacrylate, two different types of acrylic adhesives (one toughened and another one modified) and a pressure sensitive adhesive (PSA) tape. The experiments enabled to conclude that the use of a PSA tape is the best solution for achieving a strong bond and satisfactory environmental resistance. The best joint design, for the selected adhesive, was also determined by performing. mechanical testing, evaluating the joints strength, and optical microscopy, to determine the failure mechanisms. For the tested joints it was found that PEM fracture, or a combined failure mode, consisting of loss of adhesion and cohesion of the adhesive, at different extents, were the main failure mechanisms.

Resumo

Para combater as mudanças climáticas e preparar o caminho para uma economia de neutralidade carbónica, a União Europeia e os seus estados membros têm implementado estratégias que reconhecem o potencial do hidrogénio para apoiar o esforço global de atingir as metas do Acordo de Paris. Nesse sentido, é necessária inovação e otimização de sistemas que utilizem o hidrogénio como fonte, combustível ou portador de energia.

As células de combustível com membrana de permuta protónica (PEM) são um tipo de célula de combustível que converte a energia química do hidrogénio em eletricidade. Embora esses dispositivos sejam promissores para a indústria dos transportes, apresentam desafios de fabricação e fiabilidade.

A montagem das células de combustível e dos *stacks* de células de combustível disponíveis atualmente envolve apertar firmemente os componentes para garantir a vedação adequada. Os parafusos usados para manter o sistema a funcionar corretamente também apresentam problemas de corrosão com o uso. Para resolver esse problema, é considerado um projeto alternativo que descarta a necessidade desses fixadores e sistemas de compressão exigentes durante a montagem.

Este trabalho propõe uma moldura estrutural polimérica para suportar a membrana eletrolítica de permuta protónica (PEM). Esta moldura substitui o contacto vedado entre o eletrólito e as placas bipolares num projeto típico de célula de combustível. O polímero estrutural é uma poliéter éter cetona (PEEK). Uma vez que a PEM é de Nafion, o desenvolvimento de soluções para a união dos dois polímeros diferentes é importante para o desenvolvimento de um novo design de célula de combustível.

Com base no problema descrito acima, o trabalho experimental realizado no âmbito desta investigação foi planeado com o objetivo de resolver o desafio associado à ligação por adesivos dos dois polímeros. Como os dois polímeros têm baixas energias de superfície, a sua ligação por adesivos constitui um desafio de engenharia, que inclui não só a seleção do adesivo apropriado à aplicação, mas também o desenvolvimento de um design de junta.

O trabalho realizado envolveu a seleção do adesivo apropriado, entre um cianoacrilato, dois tipos diferentes de adesivos acrílicos (um reforçado e um outro modificado) e uma fita adesiva sensível à pressão (PSA). O trabalho experimental permitiu concluir que fita PSA é a melhor solução para se obter uma ligação adesiva forte e uma resistência ambiental satisfatória. Para o adesivo selecionado, foi encontrado o melhor desenho de junta, para o adesivo selecionado, através da execução de ensaios mecânicos, avaliação da resistência das juntas, e microscopia ótica, para determinação dos mecanismos de falha. Para as juntas testadas, verificou-se os principais mecanismos de falha foram a fratura da PEM, ou um modo de falha combinado, consistindo na perda de adesão e coesão do adesivo, em diferentes extensões.

Palavras-chave: Nafion, PEEK, ligação por adesivos, fita adesiva sensível à pressão, ligação de materiais dissimilares, membrana polimérica de permuta protónica.

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LIST OF ACRONYMS

Acronyms

DEM – Departamento de Engenharia Mecânica

EW – equivalent weight

FCTUC – Faculdade de Ciências e Tecnologia da Universidade de Coimbra

MD – machine direction

MEA – Membrane Electrode Assembly

MIT – Massachusetts Institute of Technology

OL – overlap length

PEEK – polyetheretherketone

PEM – proton exchange membrane or polymer electrolyte membrane

PFSA - perfluorosulfonic acid

PSA – pressure sensitive tape

PTFE - polytetrafluoroethylene

RH – relative humidity

TD – Transverse direction

1. INTRODUCTION

1.1. Motivation

At the present, there is a strong demand on the search for clean and sustainable technologies, as a gateway to meet the goals of the global warming mitigation agreements, such as the Paris Agreement [1], within the United Nations Framework Convention on Climate Change (UNFCCC). As a renewable and powerful fuel, hydrogen is deemed to take an important role in the energetic transition to a more sustainable society.

The Polymer Electrolyte Membrane (PEM) fuel cells, in which the electrolyte is a polymer membrane, are pointed as one of the most promising solutions for a variety of applications ranging from heavy-duty mobility to stationary power generation [2], [3]. PEM single fuel cells are usually a fastened compressed system with the following components: an end-plate, a current collector, a flow field plate and the Membrane Electrode Assembly (MEA) (that, in traditional systems, comprises of the polymer membrane, catalyst layers, gas diffusion layers (GDL) and sealing materials) and again a flow-field plate, current collector and end-plate [2], [4].

However, even though current fuel cell technology is proven, some issues have held back its widespread use. Besides the hydrogen infrastructure needed, the manufacturing of fuel cell stacks is a problem [5]. For optimal system performance and reliability, as well as cost efficiency, it would be desirable to integrate the assembly and sealing of the MEA components, or change the materials altogether [6], [7], [8], [9]. To tackle these issues, a novel fuel cell design has been proposed introducing a structural PEEK frame that makes the interface between the MEA and the plates of the fuel cell. This work focuses on the joining of that PEEK frame to the PEM of the MEA through adhesive bonding, as an alternative to the conventional fuel cell assembly through fasteners. The scope of this work is to develop single-lap adhesive joints that can withstand temperatures up to 80ºC, continuously or intermittently, in water or high humidity. In addition, the adhesive also needs to have the strength, in different loading modes, required for the resulting joint to withstand a flow pressure of around 3 bar. The catalyst layer applied on the PEM may also create an oxidative neighbouring area which needs to be taken into account when assessing the joint

strength. To ensure air and water tightness, the adhesive should provide sealing. Furthermore, it should be an electrical insulator to keep the current flowing appropriately. For system cost, mass and volume considerations, the adhesive should be able to perform with the least amount of material possible.

The used PEM is the state-of-the-art Nafion and because of its chemical similarities to PTFE, adhesive bonding is a challenge due to its low surface energy [10]. The PEEK frame and the PEM were bonded in single-lap joints. The joining of these two materials has not been documented yet, so different adhesives, such as acrylics, modified acrylics, cyanoacrylates and pressure-sensitive adhesive tapes were tested. Upon successful bonding using a pressure-sensitive adhesive tape was achieved, a strength analysis was carried out considering different overlap areas. The samples were also subjected to a microscopic analysis to assess the failure mechanisms.

1.2. Layout

This work is divided in six chapters. Chapter 1 provides a brief introduction and the motivation. A literature review of the materials and associated components in fuel cells is done to provide context for design considerations. This chapter also gives an overview of adhesive bonding. Chapter 3 explains the joint design. Chapter 4 describes the substrate materials, experimental methodology and used equipment. Chapter 5 presents the results for the lap-shear strength tests and microscopic observations along with their analysis. Chapter 6 closes this work with the conclusions and suggestions for future work.

2. LITERATURE REVIEW

2.1. Polymer Electrolyte Membrane Fuel Cells

Fuel cells produce electrical power by fuel oxidation on one electrode, the anode, and reduction on the cathode. On the anode there is a release of electrons that are consumed in the cathode reaction and this causes a voltage that makes current flow. In other words, the electrochemistry in a fuel cell is based on oxidation-reduction reactions, which are explained in detail in literature such as [11]. Fuel cells classification can be based on the type of fuel: hydrogen, methanol or methane. In terms of hydrogen fuel cells, the chemical reaction involves the oxidation of hydrogen, which can be achieved in different ways. This can branch many different types of hydrogen fuel cells according to the operating conditions, namely: polymer electrolyte or proton exchange membrane (PEM) fuel cells, alkaline fuel cells (AFC), molten carbonate fuel cell (MCFC), solid oxide fuel cell (SOFC), or phosphoric acid fuel cell (PAFC), according to [4], [11], [12].

PEM single fuel cells are comprised of an end-plate, a current collector, a flow field plate, a Membrane Electrode Assembly (MEA) and, again, a flow-field plate, a current collector and an end-plate [2], [4]. The MEA, in traditional systems, is comprised of the polymer electrolyte membrane (PEM), catalyst layers, gas diffusion layers (GDL) and sealing materials. These elements are tightly compressed together with fasteners to guarantee that the gaskets are sealing the system appropriately [13], [14].
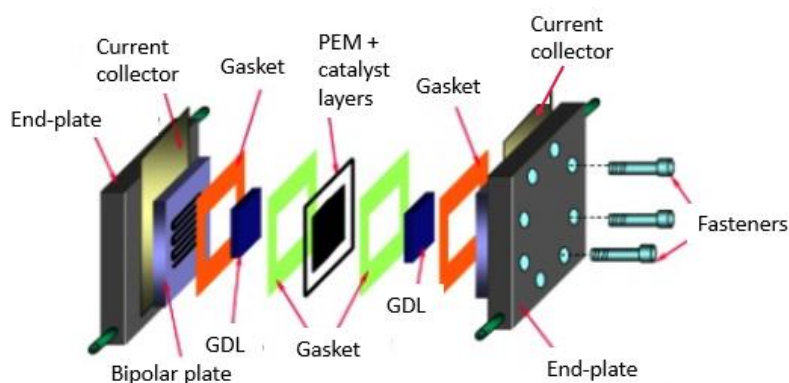
Figure 2.1. PEM fuel cell assembly schematic (adapted from [15]).

The operating pressure of PEM fuel cells can go up to 3 bar (300 kPa), although larger fuel cell systems can operate at pressures lower than atmospheric pressure [2].

2.1.1.   Polymer electrolyte membrane: Nafion™

In PEM fuel cells, polymer electrolyte membranes separate the electrodes from physical contact and prevent the fuel to cross-over between the two sides of the electrolyte (only allowing proton conductivity). Nafion™ is the state-of-the-art proton/cation exchange electrolyte membrane from Chemours[1]. These membranes are widely used not only for their unique electrochemical capabilities, but also for their excellent thermal and mechanical stability [2], [4], [11], [16], [17]. Nafion™ is the brand name for sulfonated tetrafluoroethylene fluoropolymer-copolymer. This polymer is special for its ionic properties resulting from the inclusion of sulfonated groups at the end of perfluorovynil ether groups on a tetrafluoroethylenebackbone, commonly known as PTFE[2]. It is this similarity with PTFE that grants Nafion™ the chemical inertness, which has advantages but also presents disadvantages. One, in particular, is the object of study in this work: the difficult adhesion [10]. The fluorine atoms in the chemical structure provide fluoropolymers and copolymers, like Nafion™, the "non-stick" properties [18]. Literature does not provide concrete values for Nafion™ surface energy, but it is comparable to PTFE, which has one of the lowest surface free energies for polymers with a value of 20 mN/m at 20°C [19].

In PEM fuel cells, the Nafion™ membrane should operate with low moisture content, in order to prevent problems in conductivity and fuel cell failure [4].

2.2. Structural polymeric frame: PEEK

Polyetheretherketone (PEEK) is a sub-branch of polyaryletherketone polymers. It has a semi crystalline structure that gives an excellent balance of thermal, mechanical and chemical resistance. Furthermore, it can be easily processed as a thermoplastic [18]. It is a tough and ductile polymer, with excellent fatigue properties, which can bear loads even at high temperatures, due to its high melting point (around 334ºC) and glass transition

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[1] A spin-off from the chemical company DuPont
[2] Or commercially known as Teflon

temperature(143ºC) (see ANNEX A). In terms of chemical and solvent resistance, it is inert to organics, for the most part, highly resistant to acids and bases and, also, presents high resistance to high temperature water and steam [20].

PEEK also has low surface energy (low adhesion capability) with a surface free energy lying between 34-38 mN/m. However, the surface free energy can be raised to approximately 60 mN/m with surface treatments, such as atmospheric plasma, corona discharge, flame treatment, mechanical abrasion or chemical etching [21].

2.3. Adhesive bonding of polymers

Polymers are often more difficult to join than other materials, because of their bulk and surface characteristics, which affect the surface ability to adhere to other substrates, and, in this way, the joint strength [22]. For this reason, methods such as mechanical joining, through fasteners and rivets, self-assembly or certain welding techniques are recommended in alternative to adhesive bonding [18], [22].

The adhesive is the substance that creates the bond between two materials and is typically polymeric itself. Adhesives are used for different goals. They can suitably replace mechanical fastening for their ability to distribute stresses uniformly. At the same time, adhesives can work as sealants by connecting the two surfaces very closely. Due to the typical properties of the polymeric nature of adhesives, they can also provide thermal or electrical insulation within a joint. When bonding dissimilar materials, adhesives may protect against electrochemical corrosion and damp vibrations (which is also related to fatigue resistance). Adhesives can be advantageous also for applications where aesthetics or smooth contours are important, because they can remain invisible from the exterior and be aerodynamically efficient [18], [22]–[24].

However, adhesives also display limitations and disadvantages. Generically, adhesive bonded joints need to be carefully designed and fabricated according to the operating conditions, since the success of the adhesive bond deeply relies on the surface characteristics of the materials to be joined: the surface energy of the material is preponderant. Some types of adhesives are particularly sensitive to the environment and to the combined effect of the environmental conditions with the mechanical loading. Adhesives usually can't be used for high temperature operating conditions and the surface preparation

of the elements to be joined is very important for the performance and durability of the joint. Assembly usually requires heat or pressure systems, as well as jigs and fixture systems. A strict quality control process should be in place for adhesive joints manufacturing [18], [22]–[24].

In adhesive bonding, the two materials to be joined are called adherends or substrates. H. Sharpe extended the ASTM definition of adhesive as "a substance capable of holding materials together in a functional manner by surface attachment" [24]. When applied on a substrate or adherend, adhesives wet the surfaces to form the bond, meaning that at some point while forming the bond, they must behave like a liquid. Depending on its nature, the adhesives then cure through mechanisms such as solvent loss, cooling from melt to solid, cross-linking (or polymerization chemical reactions) or pressure [23]. If an adhesive is able to cover and coat an adherend surface, the bond will happen more easily and the surface is said to be "wet". Making an analogy with liquids, when a solid surface is wetted by the liquid, it means that the surface energy of the solid is greater than the surface energy of the liquid. On the contrary, if the liquid forms beads on the solid surface, this means that the surface energy of the liquid is greater than the surface energy of the solid [18], [23], [24].

The adherend and the adhesive can be linked by strong covalent bonds. This type of bonding may be strong and durable, however it requires chemical affinity between the materials. Alternatively, hydrogen bonding, van der Waals forces and induced dipoles, also provide attraction between the adhesive and the adherend with medium strength. These bonding mechanisms are important as well for the cohesive strength of the adhesive polymers, since they provide some resistance to chain movements when the joints are loaded, avoiding that the chains simply slide, disentangle and destroy the bond. Mechanical bonding may complement the other bonding mechanisms, by allowing the adhesive to flow into surface irregularities, locking the two surfaces together, and also allowing a better stress distribution (more contact surface) [24].

The production of adhesive joints involves surface preparation, the application of the adhesive and its curing. After the surface preparation, the adhesive is applied. If the adhesive is a liquid or a paste, it can be applied by brushing, spraying, dip coating or with the help of spatulas, applicator guns or rollers. The choice of the application method usually depends on the viscosity of the adhesive, its composition (if it is a 2-part adhesive that requires mixing, for example) and on the geometry of the adherends. Dip coating and

spraying are useful for flat or contoured parts. Thin paste adhesives can be brushed. Film adhesives can be cut to size, placed and tacked. Jigs and fixtures may be needed to maintain the pressure during cure or restrict the bond area and control the bond line thickness. The curing process should start after the application of the adhesive. Pressure is usually required to help the adhesive to flow and fill in the gaps between the adherends, while keeping the adherends in the right position. The curing process is usually time and temperature dependent and may vary according to the type of adhesive [18].

The next subsections explore important considerations when designing for adhesive bonding. On a primary basis, the joint configurations and the surface characteristics that need to be considered when selecting an adhesive are discussed. An overview of the different types of adhesives available for different applications is then given. Finally, in order to assess the behaviour of the joints, an explanation on testing and failure analysis is also provided.

2.3.1. Joint configuration

Joints should be able to withstand different types of mechanical loading, such as tensile stress (normal to the plane of bond), shear stress (parallel to the plane of bond), peel stress (combination of shear and tensile) and cleavage (combination of non-uniform shear and normal stresses in the bond). Typically, adhesives display the highest and lowest load-bearing capacity in shear and peel loading, respectively. A good joint design should distribute the stresses as much uniformly as possible. Discontinuities, like edges, are stress concentration areas that should be avoided [22]–[24]. Figure 2.2 shows examples of common adhesive joint configurations.
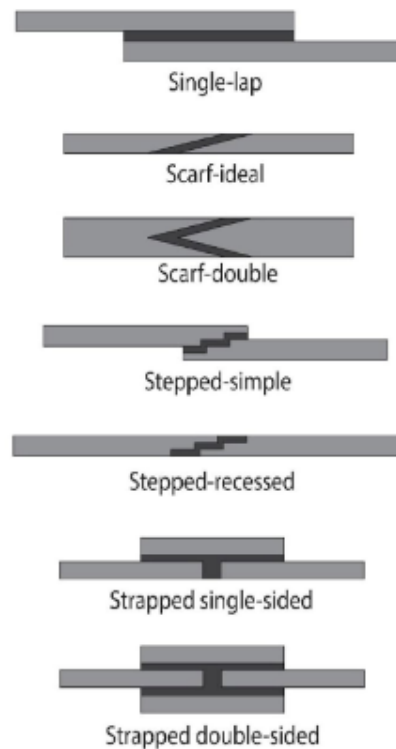
Figure 2.2. Examples of adhesive joint configurations (from [25]).

2.3.2. Surface characteristics and preparation

Adhesion is not a material property, but a combined response of the properties of the assembled materials to a destructive solicitation [24]. The surface characteristics of the adherends are the key to the success of any adhesive bonding. Depending on the wettability of those surfaces, different types of surface cleaning or preparation may be required [18].

Because adhesives work by surface attachment, surface conditions and chemistry, govern the surface energy, and therefore, adhesion. In polymeric materials, a surface can be a three-dimensional region with a few millimetres of depth over an area [22]. For the purpose of this text, surface refers to the portion of material (in depth and area) that contacts and interacts with another component or material. A plastic surface can include different constituents and boundary layers, such as moisture, migrating additives, adsorbed films or contaminants (low-molecular constituents and oxides), which are weak-links in adhesive bonding and might lead to failure. So, surface cleaning is an important first step in

adhesive bonding, even in materials with high surface energy. Contaminants at the surface interfere with the bonding process by reducing the surface energy and the contact area between the adherend and the adhesive, or by creating weak bond areas, and potential corrosion spots, where stresses may be concentrated. Wiping or spraying the surface, as well as dipping the material in a solvent, are common surface cleaning methods. The solvent must be able to remove the contaminant without reacting with the adherend surface, being easy to be rinsed from it. Vapour degreasing, for example, uses hot solvent vapours [18], [23], [24].

For some polymers with very low surface energies, simply cleaning the surface may not be enough for the adhesive to wet the surface. In order to allow the adhesive to wet the surface, some special treatments need to be carried out to increase the surface energy. Examples of these treatments are the use of primers, chemical etching, flame treatment, corona treatment or plasma treatment.

Primers are coupling agents that may be applied on the surface of the adherends, before the application of the adhesive, with the goal of producing a more uniform bond, although, in certain situations, it may also provide a stronger bond [24].

Chemical etching consists in applying a proper chemical that reacts with the adherend, providing some micro-roughness, which increases the surface energy. The etchant should be removed prior to adhesive application [22]–[24].

Polyolefins and some other plastics, such as polyethylene and polypropylene can be flame treated to oxidise the surface. Similarly, passing an electrical spark (called corona) over the surface can also promote oxidation. The corona treatment is more aggressive than the flame treatment, since it can also erode the surface besides oxidising it. Among the products of these reactions are OH and O groups that are very energetic, hence increasing the surface energy [22]–[24]. Finally, the plasma treatment requires expensive equipment and vacuum, but it can modify the surface by cleaning it, etching or reacting [22]–[24].

Mechanical abrasion is used, not because it raises the surface energy of the substrate, but because it increases the contact area between the adhesive and the substrate. Abrasion creates surface imperfections, such as roughness, cracks, pits and craters, where the adhesive can flow into, creating a mechanical interlocking with the adherend and increasing the surface area in contact, modifying the stress distribution. Mechanical abrasion does not promote any improvement in wetting, nor any thermodynamic modification of the surface, only enhances the mechanical forces helping in the adhesive bonding [24].

The choice of the surface preparation method relies on the analysis of the cost and effectiveness of the different techniques described above [23], [24].

2.3.3. Types of adhesives

There is a wide variety of commercially available adhesives, in order to fit the needs for different applications and/or subtract materials combinations. Among the criteria for selecting an adhesive are the ability of the adhesive to bond with the adherend, its compatibility (related to the effects induced on other materials, such as corrosion) with it, the cost, the joint strength provided, the environmental and chemical resistance in the service conditions, the durability, as well as other properties, such as the sealing, electrical or thermal insulation and vibration dampening properties, and process limitations (cure times, necessary fixtures and jigs, joint configuration) [23], [24].

Adhesives are commercially provided in the form of liquids, pastes, films or tapes. They can be classified in different manners, but performance and chemistry are the most common systems. A summary of the different categories is provided in the following paragraphs, and was based on the available literature [18], [22]–[24].

Structural adhesives are a category of adhesives suitable for applications in which strength and environmental resistance are crucial. They are usually made of one or two-part systems, which cure at room or higher temperatures, in order to accelerate or improve the bond characteristics. They encompass different chemical families, mostly thermosetting polymers. However, thermoplastic adhesives, like cyanoacrylates or anaerobic adhesives, are also in this category. The main advantages of the structural adhesives are their superior strength, and environmental and temperature resistance, when compared to other types of adhesives. The limitations are related to the need for proper mixing systems, for two-part adhesive formulations, and the need for heating systems, for both one-part and two-part adhesives.

Epoxies are probably the most well-known group of structural adhesives. They can be 1-part or 2-part thermoset adhesives. Meanwhile the first type is heat cured, the second type cures when the epoxy is mixed with a hardener (hence 2-part). The curing mechanism is ionic polymerization. When cured, structural adhesives are usually brittle, providinge high strength bonds (even at temperatures above 60ºC). The cost is relatively low

and there are many formulations available with different additives. When used in the joining of low surface energy plastics, structural adhesives may not be successful in providing bonding, unless expensive surface treatments are done [26].

Polyurethanes generally maintain properties at low temperature but are more unstable at high temperatures. These adhesives start curing by mixing two reactants, a polyol and an isocyanate, without need for heat, thus pot life is very short. They come at moderate cost and provide non-stiff, ductile joints at low temperatures. Even when cured, polyurethanes are sensitive to moisture and heat.

Modified acrylics are usually 2-part adhesives consisting of a modified acrylic and a surface activator. They are based on polymethyl methacrylate grafted to a rubber. When the base and accelerator parts are mixed, it activates a free-radical polymerization cure. Because of this cure mechanism, this type of adhesives can be used even when the surface preparation is not exhaustive and can provide both ductile or fragile joints.

Cyanoacrylates have a moisture cure, i.e., the liquid monomer transforms into a solid polymer in the presence of moisture, forming bond rapidly, without the need for heat or catalysts. They are expensive, and cured durability in service conditions can be poor. However, they have high tensile strengths, are typically brittle under shear loading, and its application is simple.

Anaerobics are a subset of acrylic adhesives that also cure by free-radical polymerization and are typically one-part systems. As the name might indicate, these adhesives cure in the absence of oxygen, once the air is removed from the joint by pressing the substrates against each other. Toughness and strength are moderate, providing brittle joints. Some plastics are attacked by anaerobics.

Silicones are available in either one-part or two-part systems. Atmospheric moisture is the curing agent for 1-part systems and can be acidic or non-acidic. In two-part systems, cure is achieved by catalytic action. They are thermally stable, create non-stiff, ductile joints and possess good resistance to solvents and weather. They are quite expensive and do not provide high shear strengths even in metal-to-metal joining [24].

Phenolics and urea formaldehydes are inexpensive thermosetting adhesives that can also be 1-part or 2-part. Both systems cure by condensation, creating a by-product. They are strong and present good resistance to biodegradation and hot water. Shrinkage stresses are an issue that leads to brittleness and can promote corrosion issues [24].

Hotmelts are thermoplastic solid adhesives that are applied in a molten state, using a proper heating and dispensing unit, and cure or harden by cooling off. They can form bonds very quickly and have good gap-filling capability. As thermoplastics, their temperature resistance is limited and since there is a rapid increase in viscosity with cooling, fixture time is limited. The most common chemical formulations for hotmelts are ethylene vinylacetate (EVA), polyvinyl acetates (PVA), polyethylene (PE), polyamides or polyolefins.

Pressure sensitive adhesives are tacky layers of ductile adhesives, supported in tapes or other backing material, with or without a release liner. Chemical families, for pressure sensitive adhesives, include rubbers, polyacrylates and polyvinylalkylethers. They are advantageous due to their uniform thickness, ease of application and for not requiring any activation whatsoever. The main disadvantages are their limited heat resistance and poor gap filling ability. To be able to flow and wet the adherend surface, these adhesives must have a viscoelastic behaviour, when both surfaces are pressed together. However, at the same time, they must avoid excess flowing and have some degree of elastic behaviour to provide peel and tack resistance. The adhesive polymers have high molecular weight and chain mobility, so they behave as a liquid when deformed at a low rate (or high temperatures), and they behave as a solid, when deformed at high strain rates (or lower temperatures). This happens since, at high strain rates, the chains do not have enough time to disentangle and move.

Water-base adhesives are made of compounds combined with water, like latex. Examples are starch-based adhesives, latex rubber, casein, animal glues, sodium silicate. Non-natural compounds include polyvinyl alcohol, polyvinyl acetate, amino resins, block polymers and many rubbers. Their performance is often worse than that of the other adhesives already described but they are non-toxic and non-pressure sensitive (other joint designs are allowed). They also present poor water resistance.

Radiation-cured adhesives are typically free-radical materials that cure when placed between the adherends and subjected to radiation. Electron beams and UV light are common radiation sources. They are fast cure, require no mixing and can be used with heat-sensitive substrates. However, the use of these adhesives is limited to the transparency properties of one of the substrates, besides being costly and providing poor resistance to weather, in the case of UV cures.

2.3.4. Testing of adhesive joints

Adhesive joints require testing specifically related to the application, to ensure that the bond meets the desired requirements in terms of strength, environmental resistance, and durability.

A test program for adhesive joints should start by experimenting different adhesive formulations, in order to select the one that produces the most viable solution for the envisaged application. After finding the most suitable adhesive, the test program should continue by assessing the performance of the joint, in the worst-case scenario conditions, in order to determine how it endures service conditions [22], [23].

Adhesive joints can be subject to destructive and non-destructive testing. In terms of destructive tests, peel tests, lap shear tests, or even tests tailored according to specific joint configurations may be conducted. Environmental tests, consisting of exposing the joint to solvents, thermal cycling, radiation, vibrations or other service factors, are also common. Among the non-destructive tests, proof-loading to the target loading requirement, ultrasonic testing and other sorts of visual inspection can be useful. Despite non-destructive testing is not adequate to control the bonding strength, it provides indicators on performance issues [18].

Single-lap joints are very common in adhesive testing, because under shear loading, they develop a non-uniform stress distribution along the lap which causes peak stresses at the ends of the overlap. This behaviour is explained by the varying strain between the adhesive and adherends, as well as the load eccentricity that bends the joint (because of the thickness of the joint itself). This bending moment bends the adherends materials and induces normal stresses at the ends of the lap length. The shear strain and stress will peak at each end of the overlap in the length direction and be a minimum at the middle, and if the adherends are made of different materials, the adhesive shear stress distribution will be asymmetric. In plasticity, there more effects to consider. Premature failure can happen due to the straining differentials within the adherends (that lead to increased stresses on the adhesive) [24].

2.3.5.   Failure analysis

Upon adhesive bond failure, a forensic investigation should be conducted to assess the cause of the failure. It should be determined if the joint was subjected to adverse environmental conditions or loads that did not meet the design specifications. The type of failure needs also to be determined. This will help in understanding whether failure was promoted by an isolated problem or by service conditions, and, in this way, to determine whether the problem can or not be solved. The investigation should include a review on the preparation of the joint, application of the adhesive and curing procedures, to determine whether any important step in joint production was disregarded. Ideally, in adhesive bonding, the substrate should be the weakest link of the joint [18], [23], [24].

There are four modes of adhesive bond failure to be considered: cohesive, adhesive, interfacial and mixed. Cohesive failure may occur within the adhesive system, in the oxide layer or in the substrate. This mode of failure indicates that the joint is operating at the ultimate performance and the only margin for improvement is replacing the weakest link or redesigning the joint. Adhesive failure occurs between the adhesive or primer, and the adherend, by loss of adhesion, and it means that the bond can be strengthened by improving the interface. Interfacial failure is similar to adhesive failure, but rupture occurs by separation of a thin oxide layer (or a contaminant layer) from the adherend. Mixed-mode failure is a combination of the above-mentioned failure modes [23], [24].

Cohesive failure   Adhesive failure   Interfacial failure

Figure 2.3. Adhesive bonding failure modes: a) Cohesive failure; b) adhesive failure; c) interfacial failure (adapted from [27]).

As it can be concluded from the analysis of the different failure modes, in adhesive bonding, the strength of the joints depends on factors such as the adherend properties, the distribution of voids in the contact interfaces and boundary layers, the joint geometry and the loading mode itself [24].

Visual inspection is useful and may be sufficient to determine failure mode in some cases. Microscopy techniques can also be used for forensic analysis, depending on the available instruments, the resolution required and the nature of the surface [24].

3. OBJECTIVE OF CURRENT WORK

3.1. Joint design

The goal of this work is to produce an adhesive bonded joint between a PEEK frame and a state of the art Nafion PEM, as shown in Figure 3.1. For better comprehension, the membrane will be always referred to as "PEM" from now on, except when referring specifically to the Nafion material properties (in which case it will be referred to as Nafion). The PEEK frame comes up as an interface component with the bipolar plates. The joining of the PEEK to the bipolar plates is pre-defined and out of the scope of this work. The PEM and the PEEK should be adhesive bonded in order to get assembly and sealing through the same process. For the intended stage of development, the joint concept illustrated in Figure 3.1 can be translated into a single-lap joint. According to the planned joint configuration, illustrated in Figure 3.2, the adhesive is spread or applied on the PEEK surface and the PEM is placed to adhere to the other surface side of the adhesive, bonding the two substrates together. For a perfect control of the thickness of the adhesive layer, a groove was made in the PEEK surface. The depth of the groove was equal to the intended adhesive thickness.
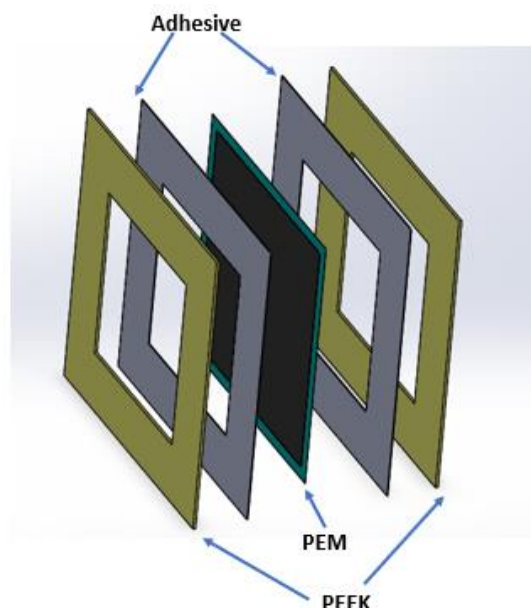
Figure 3.1. PEM to PEEK frame adhesive joint for the fuel cell system.

For the joint design envisaged, the adhesive properties may be summarized as follows:

- Be able to bond low surface energy materials;
- To have high strength (resulting joint should withstand around 3 bar);
- To operate at temperature up to 80°C (continuously or intermittently);
- To operate in an aqueous or moist environment (and also proximity to potentially oxidative environment);
- To provide sealing;
- To be an electrical insulator;
- To provide a bond line thickness as thin as possible.
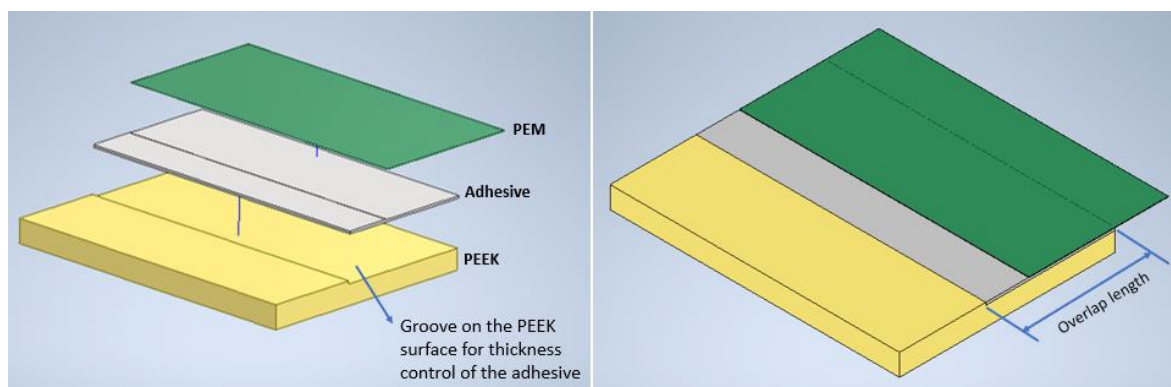
Figure 3.2. Joint design concept: exploded view (left) and assembly (right).

3.2. Selection of adhesives for experimental work

There are many adhesive products and brands on the market that meet varying needs at different prices. From a theoretical point of view, very few commercially available adhesives can be potential candidates.

Adhesive datasheets do not provide specific data concerning the adhesion properties in PEEK or Nafion™ joints. Although, some data is available for PTFE, which was used as reference, in this work, due to the similarity of this polymer with Nafion™. After an extensive adhesive data analysis, four adhesives were selected to be tested for the envisaged application, based on the described requirements:

- Cyanoacrylate adhesive meant for plastic and rubber substrates;
- Toughened acrylic adhesive for low surface energy dissimilar substrates;
- Acrylic-based adhesive meant for low surface energy surfaces;
- Double coated PSA tape specific for low surface energy surface.

The main characteristics of these adhesives are summarized in Table 3.1. Additional information is provided in ANNEX B, where the datasheets from the adhesive suppliers are provided.

Table 3.1. Summary of the characteristics of the pre-selected adhesives.

In-text reference	Characteristics	Commercial product	Temperature range
Cyanoacrylate	Cyanoacrylate Liquid adhesive	**LOCTITE® 406™ + primer LOCTITE® SF 770™** [28]	Up to 120ºC
Permabond acrylic	2-part adhesive 1:1 volume mixing ratio toughened acrylic adhesive (methacrylate)	**PERMABOND® TA4610** [29]	Up to 80ºC
3M acrylic	2-part adhesive 10:1 volume mixing ratio acrylic-based adhesive	**3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue** [30]	Up to 80ºC
PSA tape	double coated acrylic foam pressure sensitive adhesive (PSA) tapes	**3M™ VHB™ Tape LSE-060** [31], [32]	Up to 150ºC in short-term and 90ºC in long-term

4. METHODS

4.1. Adherend materials

4.1.1. Nafion™ N115

Nafion™ is the state of the art for PEM fuel cell membranes. Nafion™ membranes are a whole family but, for this work, Nafion™ N115 was used since it is rather cheap, without the need to order large quantities, and readily available online from different stores supplied by Chemours.

Nafion™ N115 is an unreinforced PFSA (perfluorosulfonic acid) membrane with 127 μm thickness, chemically stabilized, permeable to protons and can absorb water. Since water absorption decreases the Nafion™ thermal stability, the operating temperature of the membranes is more limited by the requirement to be wet, than by the material's glass transition or melting temperatures [33]. It is a fragile product that requires care, so environment-controlled storage conditions should be provided, i.e., shielding from sunlight and control of relative humidity and temperature need to be provided in order to avoid dimensional changes [33]. As previously discussed, Nafion™ has very poor adhesion properties. There is no evidence in literature of successful surface preparation for adhesive bonding, however its acidic chemistry can interfere with surface treatments that act on a chemical level.

A summary of Nafion™ N115 properties, provided by the manufacturer, is presented in Table 4.1. The mechanical properties are provided in the Machine Direction (MD) and Transverse Direction (TD).

Table 4.1. Nafion™ N115 properties at 50% relative humidity (RH) and 23ºC; MD stands for Machine Direction and TD stands for Transverse Direction [34].

	Nafion™ N115 (properties at 50% RH, 23ºC)
Thickness [µm]	127
Tensile Modulus [MPa]	249
Maximum Tensile Strength [MPa]	43 in MD; 32 in TD
Elongation at Break [%]	225 in MD; 310 in TD
Thickness increase from 50% RH, 23ºC to water soaked, 23ºC [%]	10
Linear expansion from 50% RH, 23ºC to water soaked, 23ºC [%]	10

4.1.1.1. Procedure for tensile testing of Nafion™ N115

Tensile tests were done on the product, to assess any possible change in mechanical properties during the storage in the laboratory. The Nafion™ N115 specimens were designed following guidelines for specimen Type II from ISO 527-3 "Plastics – Determination of tensile properties – Part 3: Test Conditions for films and sheets" [35]. The specimen geometry is shown in Figure 4.1. The small length of the specimen was selected in order to conciliate the high elongation at failure, that this material typically exhibits, with the maximum grip displacement allowed by the tensile testing equipment.

According to the manufacturer recommendations, the dry Nafion™ N115 was left horizontally in room temperature conditions, for 24h, to make sure that the specimen dimensions would stabilize with the relative humidity and temperature conditions. This procedure was also used in preparing Nafion™ N115 samples for other experiments. The geometry was marked on the sheets with a marker, in order to obtain five samples for each testing direction: machine or film extrusion direction (MD) – specimens MD01, MD02, MD03, MD04 and MD05 - and transverse direction (TD) – TD01, TD02, TD03, TD04 and

TD05. Afterwards, the specimens were cut to geometry with a surgeon knife. To ensure clean cutting, nitrile gloves and cleaning were used.
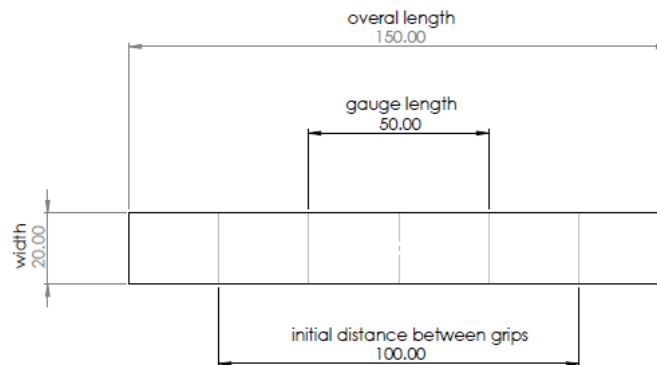
Figure 4.1. Tensile test specimen geometry for Nafion™.

Based on ASTM D882-02 [21] guidelines for an elongation at break greater than 100%), the tensile tests were conducted in a Zwick/Roell Z020 machine, at a speed of 500 mm/min. The laboratory was at 23.6±0.1 ℃ and 37±1% RH. Given the dimensions of the specimen and the very high ductility of the material, it was not possible to use an extensometer and samples elongation was assessed by the displacement of the grips.

4.1.2.   PEEK

The PEEK adherend, supplied by Röchling, was a variant, with no reinforcement called SUSTAPEEK. The material was acquired in 5mm thick plates. A summary of PEEK properties, provided by the manufacturer, is presented in Table 4.2. Those properties are very similar to other standard PEEK products, meaning that its adhesion properties should be very similar, and the surface energy should be between 34-38 mN/m [36]. Additional information on the PEEK properties is provided in ANNEX B, where the datasheet from the adhesive supplier is provided.

Table 4.2. SUSTAPEEK properties.

	SUSTAPEEK
Thickness [mm]	5
Tensile Modulus [GPa]	4
Yield Stress [MPa]	110
Elongation at Break [%]	20

4.2. Surface cleaning and preparation

PEEK was cleaned with isopropanol, abraded, cleaned again with isopropanol, and rinsed, achieving good adhesion with this simple but effective surface cleaning, for all the adhesives experimented [24]. Meanwhile, for the PEM, the chosen cleaning procedure was rinsing with deionized water and leave to dry for 24h, according to manufacturer indications [33]. The cleaning and adhesive application were conducted at room temperature and humidity conditions, that is around 23ºC and 30-50% RH.

4.3. Adhesive application

4.3.1. Cyanoacrylate adhesive

For the fabrication of joints with this cyanoacrylate (LOCTITE® 406™), after PEM surface cleaning, some of the PEM samples were also brushed with the recommended primer LOCTITE® SF 770™. The PEM started wrinkling immediately after applying the primer, which made difficult the assembly and pressure application. Excessive wrinkling was related to primer absorption and low thickness of the membrane. Other PEM samples were used without any primer at all to assess if the primer would be effective.

The adhesive was applied on the PEEK surface [11]. A small droplet, 3mm wide, was enough to cover the slot fixture. Since this adhesive has a fixture time[3] of approximately

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[3] Time until functional or handling strength is achieved

10 seconds, application needed to be precise and quick. After the application, the joints were clamped during the full 72h curing process.

4.3.2. PERMABOND acrylic adhesive

In order to determine the best procedure for the constructions of the joints, the methacrylate adhesive was applied, in a continuous bead line, on the PEEK surface, for some attempts, and on the PEM surface, for other attempts. In some trials, grooves of 0.2, 0.3 and 0.5 mm were milled on the PEEK surface to create a mechanical fixture that controlled the bond line thickness. The PEM was then assembled to the PEEK, and the joint was clamped at different pressures and different temperatures for 48h (at room temperature or inside an oven).

4.3.3. 3M acrylic adhesive

3M™ DP8010 Blue was applied in a continuous bead line, carefully, in order to avoid entrapping air, on the PEEK surface, for some attempts, and on the PEM surface, for other attempts. Again, for some of the attempts, groves of 0.2, 0.3 and 0.5 mm were milled on the PEEK surface to create a mechanical fixture that controlled the bond line thickness. The PEM was assembled, and the joint was clamped with different pressures and different temperatures for 24h (at room temperature or inside an oven).

4.3.4. Pressure sensitive adhesive (PSA) tape

After surface preparation, the desired tape overlap dimensions were marked on the PEEK surface. A piece of PSA tape was tightly stretched and applied on the PEEK surface with the help of a squeegee hand applicator to keep the tape aligned, avoid air bubbles and ensure firm pressure and get tack. The portions of PSA tape that had been touched were discarded. The edges were trimmed with a surgeon's knife, when required, to avoid stress concentrations [24]. The protective liner was then removed, carefully, from the edge aligned with the PEEK edge, in the overlap direction. This was done with the help of the tip of a surgeon's knife, in order to avoid damaging the corners of the tape. The PEM

film was attached with the help of the hand applicator to avoid entrapping air, as shown in Figure 4.2. The assembly was clamped for 72h, until full strength at room temperature was reached.
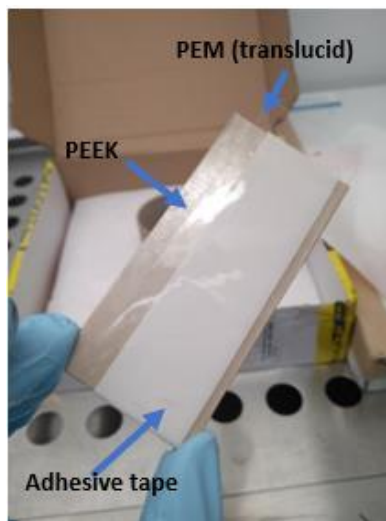
Figure 4.2. PEM to PEEK joint with 3M™ VHB™ Tape LSE-060.

4.4. Lap shear strength tests

The lap shear strength tests were carried out on single lap joint specimens assembled with the PSA tape (only), as that schematized in Figure 4.3. The testing procedure and specimen design was based on ASTM 1002–10 [37], but adapted taking into account that the substrates are polymers with very different thicknesses. Specimens with different overlap lengths (OL) were tested. In each joint, the PEEK substrate was a 13x130 mm rectangle with 5 mm thickness and the PEM was a 90x25 mm rectangle cut in the MD. The overlap width was 13 mm (the cross-section was the same for the specimens). The reason for this difference between the width of the substrates is because there is a need to have similar cross sections, since the PEM is too thin compared to the PEEK. A picture of some assembled specimens is shown in Figure 4.4.
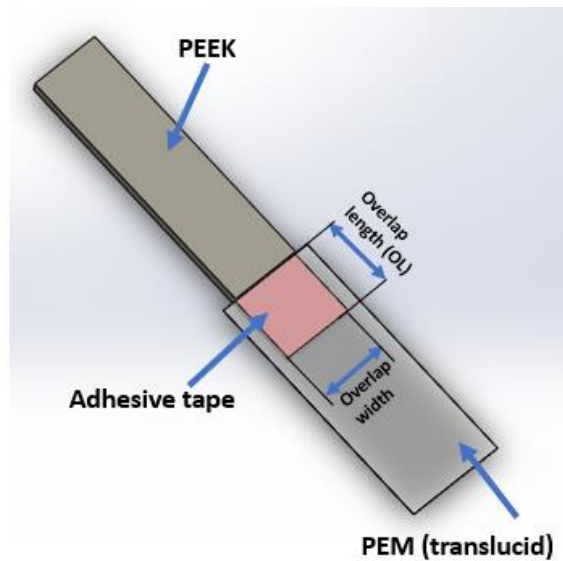
Figure 4.3. Single-lap schematic.

Figure 4.4. Single-lap joint picture.

The specimens were produced by the author. The tensile tests were carried out at 24 ºC and 37% RH, with a testing speed of 500 mm/min in a Zwick/Roell Z020 machine. A spacer was used to clamp the PEM, so that the load would be aligned with the adhesive.

4.4.1. Effect of clamping pressure during cure

The influence of the clamping pressure during cure, on the joints' strength, was assessed by testing lap-shear samples with different overlap lengths. Table 4.3 shows the test matrix, where one sample was used for each combination of parameters.

Three clamping force levels were used to produce specimens:

1. f=0 kg equivalent to no clamping pressure after the initial squeegee assembly pressure;
2. f=3 kg weight on the joint, which is equivalent to pressure values from 107 up to 235 kPa, depending on the OL.
3. f=6 kg of weight, which is equivalent to pressure values from 214 up to 470 kPa, depending on the OL.

The overlap length (OL) values were 5, 8 and 11 mm.

Table 4.3. Test matrix to study the influence of the clamping pressure during cure.

Test/Parameter levels	Overlap length (ol) [mm] - A	Pressure during cure in weight (f) [kg]- B
1	5	0
2	5	3
3	5	6
4	8	0
5	8	3
6	8	6
7	11	0
8	11	3
9	11	6

4.4.2. Effect of overlap length on joint performance

Considering the performance of the adhesives cured under 3kg force, another design of experiments was planned, in order to better assess the effect of the overlap length on the joint strength. In these tests, the specimen design was the same described above, only the overlap length was changed, assuming values of 6, 7, 8 and 9 mm.

Table 4.4. Test matrix to evaluate overlap length (OL) effect on the joint performance.

Test/Parameter levels	Overlap length (ol) [mm]
10	6
11	6
12	6
13	7
14	7
15	7
16	8
17	8
18	8
19	9
20	9
21	9

4.5. Optical microscopy

Forensic analyses were performed, on the tested specimens, in order to identify the failure modes in the different adhesive joints. This analysis was carried out using Leica / Wild M420 APOZOOM Macroscope, Nikon Epiphot 200 microscope and Nikon DS2Mv camera. The overall joint geometry, as well as some details of the corners and edges, were analysed. The specimens were observed as is, without any preparation. Observing the PEM by optical microscopy was not possible since this material is transparent to light. Observations were performed on the joints surface, along the overlap area between the adhesive and the adherends and on the cross-section of the joint, where it is possible to observe the PEEK and the PSA (and the PEM in the situations where the PEM fractured instead of peeling or losing adhesion). These observation directions are illustrated in Figure 4.5.
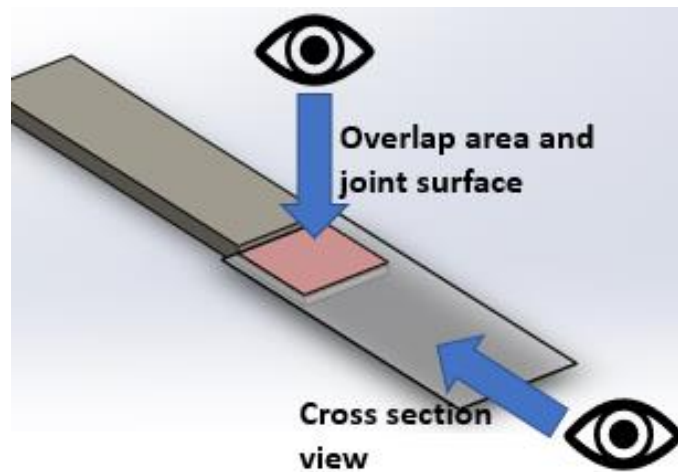
Figure 4.5. Optical microscopy view orientations.

In 5.6 Optical microscopy analysis when describing the forensic analysis results, "adhesive" and "adhesive layer" will refer to the actual adhesive coating on the surface of the supporting (or backing) acrylic foam structure of the tape. PSA or generally "tape" will refer to the combination of the adhesive coating and the support foam of the pressure sensitive adhesive tape (PSA), referring to the whole structure in between the adherends.

5. RESULTS

In this chapter the results and analyses for the experimental procedures explained in the previous chapter are presented. It begins with the mechanical characterization of Nafion. A summary of the visual inspections for the obtained joints for the different adhesive products tested is provided in 5.2. Only the pressure sensitive adhesive (PSA) tape achieved a successful adhesive bond, so the following subchapters refer to its geometrical features (subchapter 5.3), the analysis of the effect of the clamping pressure and overlap length (OL) on the mechanical performance of the joints (subchapters 5.4 and 5.5). At last, the optical microscopic analysis is presented to aid the study of the failure modes of the tested joints, in subchapter 5.6.

5.1. Mechanical characterization of Nafion™ N115

The raw force-displacement data, for the two testing directions, is presented in Figure 5.1. The figure shows that, although the mechanical behaviour in tensile loading was similar for all the specimens tested, the maximum elongation until failure was different. The same type of results was observed for the two testing directions. The increase of the stress with the elongation, is related to a work-induced increase in crystallinity of the polymer. The difference in ductility, between the samples tested in each loading direction, could be related to the presence of micro-cracks introduced during the cutting and trimming of the specimens.

The average engineering tensile strength values, for each testing direction, are compared in Table 5.1. The difference in values between the two directions is related to the manufacturing process of the Nafion membranes: Nafion is extruded into thin films, which creates anisotropy in the material. The tensile strength is higher in the extrusion direction (MD). Given the viscoelastic behaviour of Nafion [37], which is enhanced with water intake [38], the tensile strength values are more critical than the yield strength values [38].
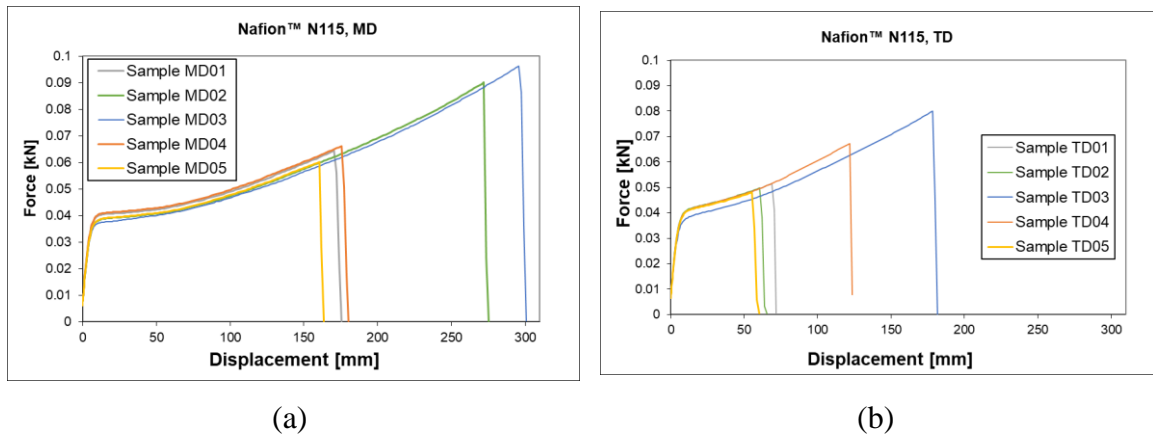
(a)　　　　　　　　　　　　　　　　(b)

Figure 5.1. Force-displacement graphs: (a) Machine Direction; (b) Transverse Direction.

Table 5.1. Nafion™ N115 PEM tensile strength.

	MD	**TD**
Engineering Tensile Strength [MPa]	29.70 ± 6.51	23.88 ± 5.46

5.2. Adhesive joints inspection for the different adhesive products

After the curing was completed, all the adhesive joints were visually inspected. The visual inspection enabled to conclude that the joints bonded with the cyanoacrylate adhesive, the PERMABOND acrylic and the 3M acrylic adhesives had important defects, indicating that the different levels of heat and pressure applied for curing could not create a bond. In contrast, for the PSA tapes, no important defects were detected, and, for that reason, the joints made with this adhesive were subjected to further tests. Jules E. Schoenberg [39] stated that acidic surfaces are poor at initiating cyanoacrylate polymerization. Given the acidic chemical nature of the Nafion^{TM} adherend, this is a plausible explanation for the poor quality of the bonding with those adhesives However, no explanation was found on why the acrylic pressure sensitive adhesives (PSA) worked and the other acrylic based adhesives did not, the major difference being the underlying curing mechanism (details on the chemical compositions of the adhesives are trade secrets).

5.3. The pressure sensitive adhesive (PSA) tape

This pressure sensitive adhesive (PSA), supplied in the form of a tape, was analysed using optical microscopy. The tape is comprised of a backing acrylic foam with an adhesive film coating it on each side (hence double coated tape). Figure 5.2 shows the cross section of the PSA tape without the protective release liner. The PSA tape was 0.6 mm thick, meaning that each adhesive film layer was 0.1 mm thick and the foam backing 0.4 mm thick. The micrograph of the foam layer, in Figure 5.3 (a), enables to observe that the foam had a closed-cell structure, meaning that it is a flexible structure made of internal pores that are very close to each other, but not interconnected. The size of these pores varies between 10 to 70 µm. The micrograph of the adhesive layer, shown in Figure 5.3 (b), shows a plain surface with some pores.
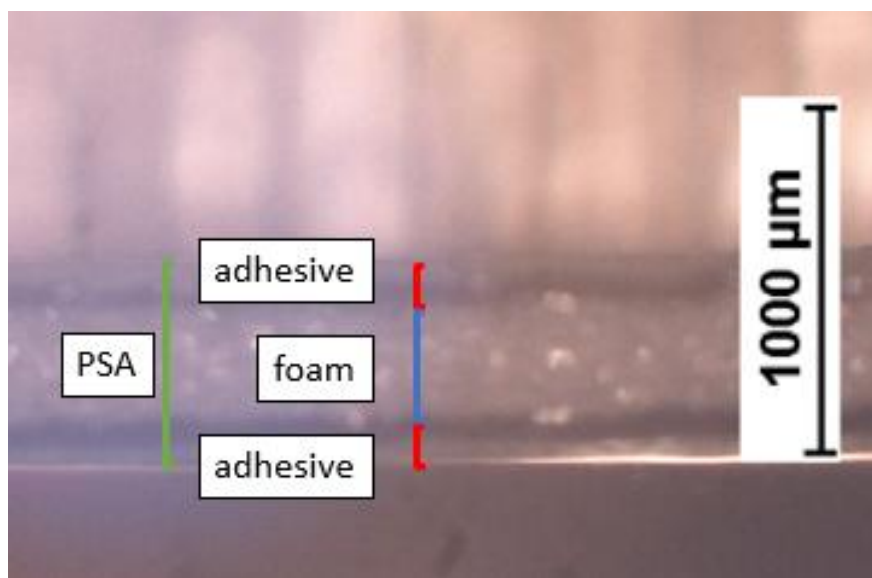

Figure 5.2. 3M™ VHB™ LSE 060 tape structure as seen from the cross section.

(a)   (b)
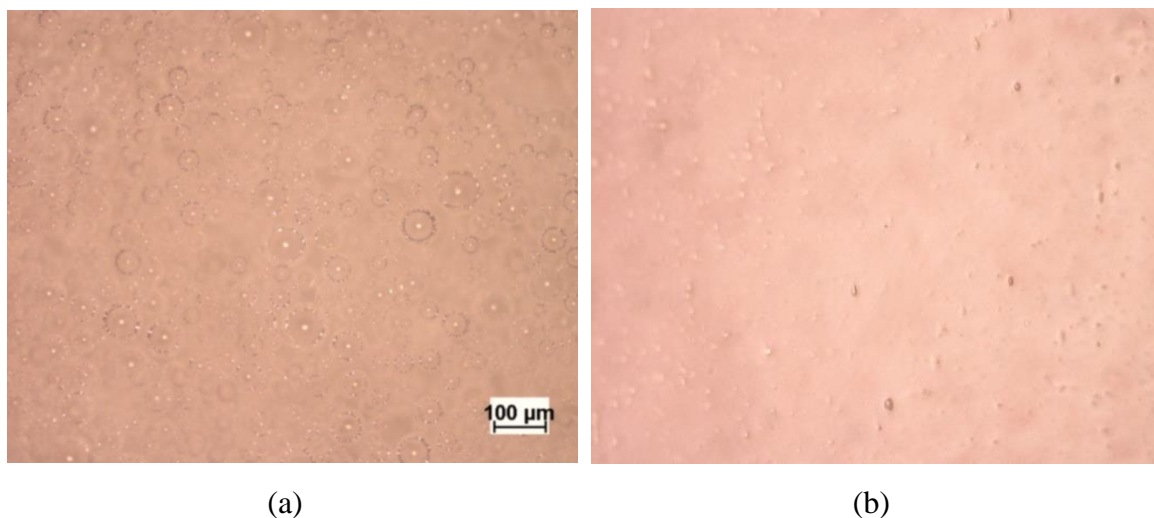
Figure 5.3. 3M™ VHB™ LSE 060 tape structure as seen from the surface: (a) backing foam; (b) adhesive coating.

5.4. Effect of clamping pressure

The force-displacement curves for the specimens of joints produced using different clamping pressures during curing are compared in Figure 5.4. Analysing the figure it is possible to conclude that the joints which were subjected to a 3kg load displayed higher strength and elongation at failure than the ones subjected to 0 and 6kg load. Applying pressure plays an important role in pressure sensitive tapes bonding mechanisms, since pressure promotes intimate contact between the surfaces to be bonded and makes it possible for the adhesive to wet the substrates [23]. Based on this, it was expectable that applying no pressure at all during cure would negatively affect the performance of the joint. Current results also show that too much pressure, is also inconvenient. According to [40], excessive pressure inhibits the flow of the adhesive, a phenomenon known as "glue starvation"[40].
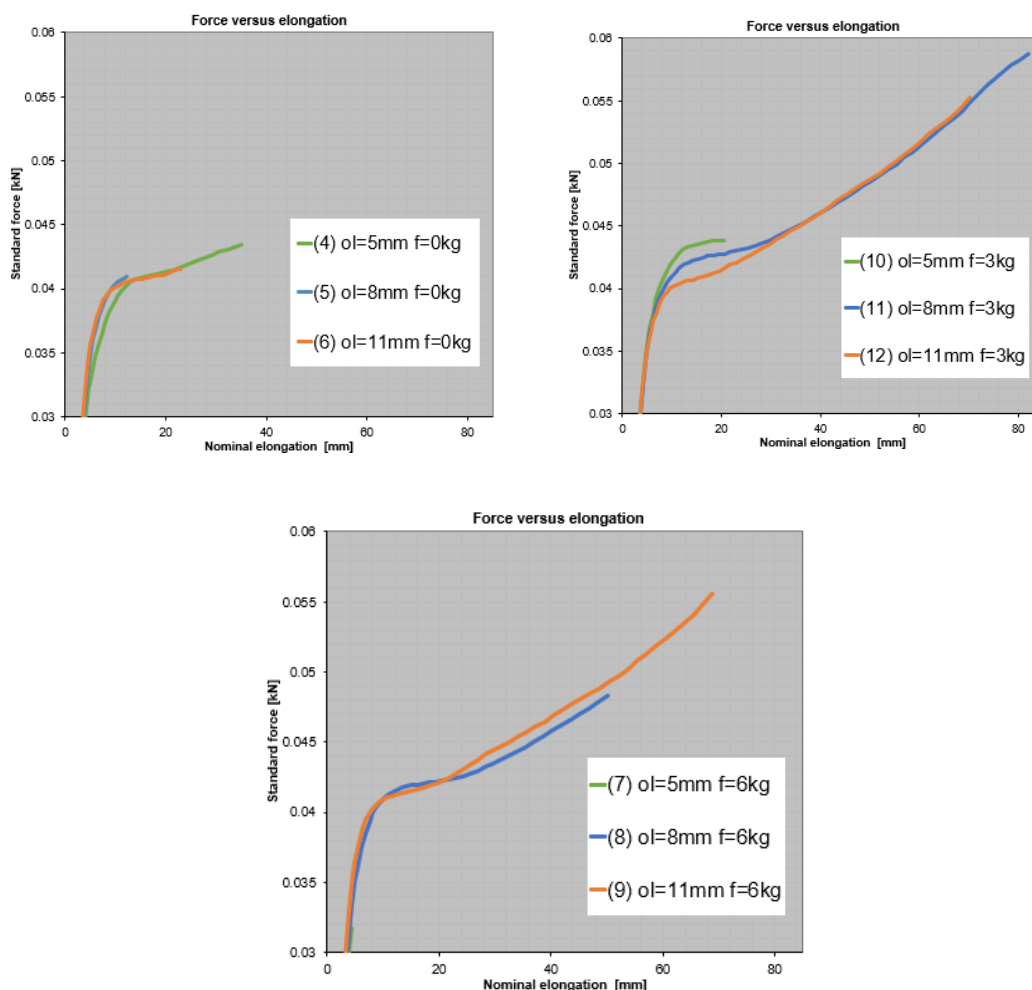
Figure 5.4. Force versus elongation plots for different overlap lengths at different curing pressures: a) f= 0 kg; b) f= 3 kg; c) f=6kg; in the legend, the number between brackets refers to the specimen number, followed by the respective parameters.

5.5. Effect of overlap length on joint performance

Figure 5.5 shows the force-displacement plots for the tested specimens, which characteristics are described in Table 5.2. In the figure, it is possible to see that the joints with the higher overlap length were the ones able to withstand the larger amount of strain before failure. These differences in the strain of the PEM can be related to the cutting and trimming of the PEM that causes micro-cracks and that is very difficult to control. However, the figure also shows that the elastic behaviour was similar for all the samples, in spite of the different overlap lengths.

Figure 5.6 shows pictures of the samples after lap-shear testing. From the analysis of the pictures, it is possible to identify whether the failure occurred at the interface between the PEM and the adhesive or it was a PEM fracture. The results of these observations are listed in Table 5.2. Although some samples showed PEM fracture instead of adhesion failure between the PEM and the adhesive, the main failure mechanism was loss of adhesion between the PEM and the adhesive, independent of the overlap lengths considered, as summarized in Table 5.2.

When the failure mode was PEM fracture, the shear strength was disregarded for the calculation of the average shear strength, for each overlap length level. The average shear strengths are shown in Table 5.2. The table shows that a joint with an overlap length of 6 mm can already provide a shear strength of over 400 kPa, which is already above the established 3 bar of internal pressure predicted for the operation of a fuel cell.
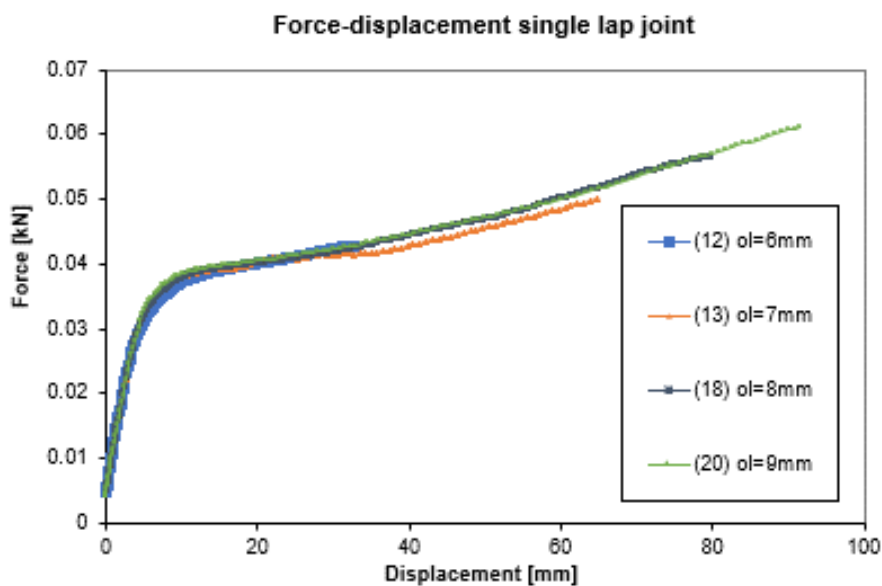
Figure 5.5. Force versus displacement plots for different overlap lengths – representative specimens.

Table 5.2. Overlap length (OL) effect on the joint performance results.

Test/Parameter levels	Overlap length (ol) [mm]	Failure mode	Shear strength [kPa]	Average shear strength [MPa]
10	6	PEM/adhesive failure	552.2	498.6±24.7
11	6	PEM/adhesive failure	393.5	
12	6	PEM/adhesive failure	550.0	
13	7	PEM fracture	-	430.3±24.4
14	7	PEM/adhesive failure	479.1	
15	7	PEM/adhesive failure	381.6	
16	8	PEM/adhesive failure	385.8	437.3±25.56
17	8	PEM/adhesive failure	380.4	
18	8	PEM/adhesive failure	545.7	
19	9	PEM/adhesive failure	399.9	461.7±30.9
20	9	PEM/adhesive failure	543.4	
21	9	PEM fracture	-	

(a) OL=6mm
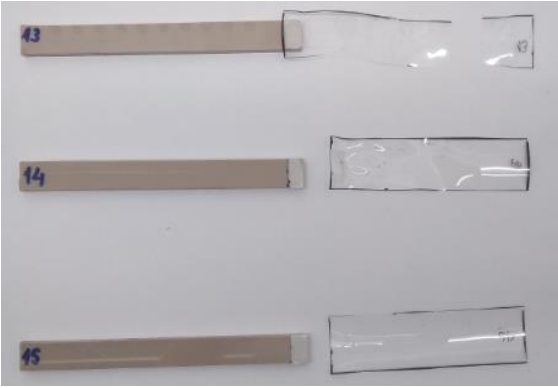
(b) OL=7mm

(c) OL=8mm

(d) OL=9mm

Figure 5.6. Specimens after the lap shear test.

5.6. Optical microscopy analysis

Figure 5.7 shows the three types of failures found in the specimens in a macro level: (I) loss of adhesion with adhesive cohesion failure; (II) cohesive failure of the adhesive and the backing foam; (III) PEM fracture. Figure 5.7 (a), (c) and (e) are top views of the adhesive tape still sticking to the PEEK surface. Figure 5.7 (b) and (d) show the matching PEM surface. Figure 5.7 (e) shows the translucid PEM, adhesive tape on top of the PEEK surface.
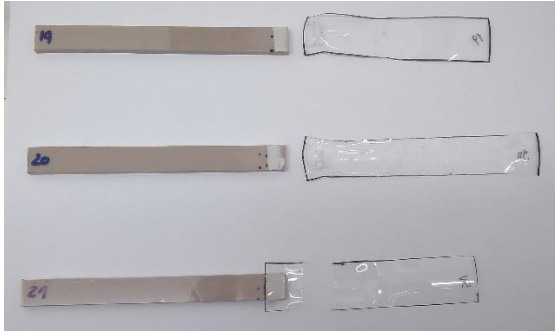
Specimen 16, corresponding to a joint with overlap length of 8 mm, is pictured Figure 5.7 (a). The image shows that this specimen experienced Failure Mode A, i.e., it is possible to see the abraded PEEK surface and the PSA. In Figure 5.7 (b), it is shown the conjugate PEM surface (the black markings are ink from the geometry marking for cutting and assembly). Similar results were reported for samples number 17, 18 and 19. The analysis of failure surface enabled to conclude that there was loss of adhesion, between the PEM and the adhesive, but also, some cohesive failure of the adhesive film, on the edge in line with the PEEK substrate edge, within the overlap area. Most of the adhesive layer seems to have stuck to the backing foam, but there is a linear remnant of adhesive on the PEM surface that is evidence. The shear stress peaks, at the ends of the overlap area, in the length direction, previously discussed in this dissertation, are evidenced in the images by this clear overload of the adhesive layer at the edge. This same edge was the one from where the tape liner was removed, which also contributed to make this area a weak link.

A more severe case of the previous situation is shown in Figure 5.7 (c) and (d), where it is illustrated the Failure Mode B for specimen 14, with an overlap length of 7 mm. In Figure 5.7 (c) it is possible to distinguish a more intense white structure on the overlap, which matches a clean transparent area in (d). This is basically the previous situation but with a wider failure area and a stronger cohesive failure, both in the adhesive layer and in the foam backing, to some extent. At naked eye, the area where there seems that the adhesive has been removed is matt, whereas the area where there just seems to have occurred adhesion failure (with the adhesive layer sticking to the backing foam of the tape) is shiny. In the image, the PEM is distorted because the strain was higher than that of the sample in Figure 5.7 (Failure Mode A). Since similar failure behaviour was registered for the samples number 10, 11, 12, 15 and 20, it could be concluded that this failure mode was independent of the overlap length. This type of failure may have resulted from improper surface conditions on

the PEM side, introduction of contaminants prior to adhesive application or because the adhesive was not able to wet the PEM properly for other unknown reasons.

Finally, Figure 5.7 (e) represents the Failure Mode C, registered for the specimens 13, with an overlap length of 7 mm, and 21, with an overlap length of 9 mm. In Table 5.2, this failure mode was described as "PEM failure". The picture shows the abraded PEEK, on the lower plane, the adhesive tape in the middle, and the PEM on the top. It is possible to see a circular feature in the image that suggests that the corners and edges of the joint area were weaker than the centre. In this case, there was a slight adhesion failure between the PEEK and the adhesive, in the corners, as evidenced by the PEEK brownish edges that should be covered with PSA. The PEM fractured close to the joint, which could mean that the bond was stronger than the substrate material. However, what might have happened, was that the PEM started bearing more load than the joint at some point. This could be due to substrate material defects or issues when cutting the specimen, which promoted stress concentrations or load misalignment (0.1 mm misalignment would be enough).

Failure Mode A – Loss of adhesion and cohesive failure of the adhesive

(a)   (b)
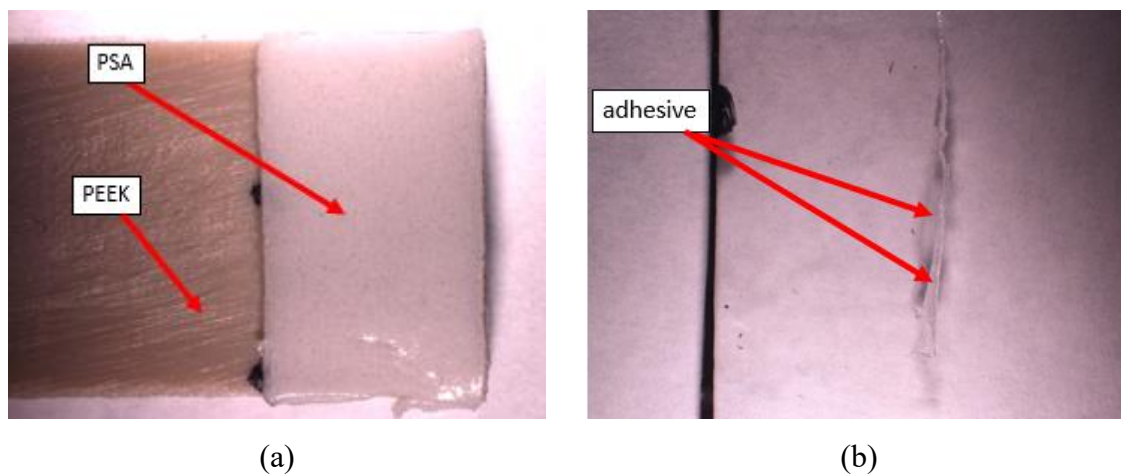
Failure Mode B – Cohesive failure of the adhesive and the backing foam of the tape

(c)   (d)

Failure Mode C – PEM failure

(e)
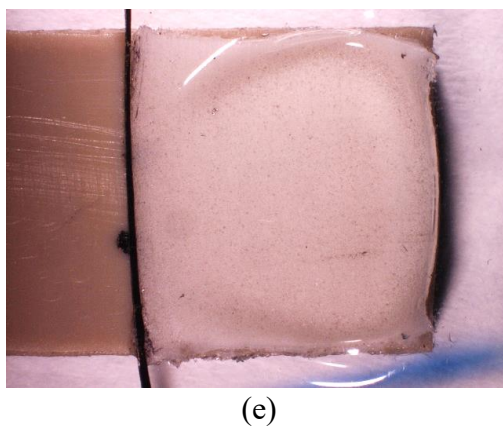
Figure 5.7. Macro pictures to identify failure modes. Situation I – adhesive cohesion failure: (a) and (b), Situation II – adhesive and foam cohesion failure (c) and (d), Situation III - PEM failure (e)

5.6.1. Failure Mode A

For specimens which experienced adhesive failure at the edge, the top surface microstructure is as shown in Figure 5.8. In detail 1, where a magnification of the edge where cohesive failure of the adhesive occurred is shown, it is possible to observe the presence of adhesive in the upper-right corner of the image (brighter area with no visible voids). Contrary to this, the foam structure, in the rest of the image, indicates that the adhesive layer was missing in that location of the failure surface. In detail 2, corresponding to the opposite overlap end, the adhesive layer is visible all the way. Detail 3 shows the same on the same edge of 2, but at opposite width side. In detail 4, the loss of adhesive and maybe some foam, is evidenced, as predicted from the macrostructural analysis. In this image is also possible to see a fibre contaminant, probably from the laminate gloves used to handle the specimens. Details 5 and 6 show the transition between a thinner lost section of adhesive and a thicker one, as the observation moves towards the middle of the edge. These images suggest that the loss of adhesion, at the adhesive-PEM interface, could be related to inadequate surface conditions. Since no major contamination was found, the loss of adhesion should be related to insufficient wetting of the surface by the adhesive. Actually, Figure 5.9 shows that, on the PEEK edge plane, there was still some adhesive from the adhesive-PEEK interface, but further away, there was foam and the PEM-adhesive layer was missing.
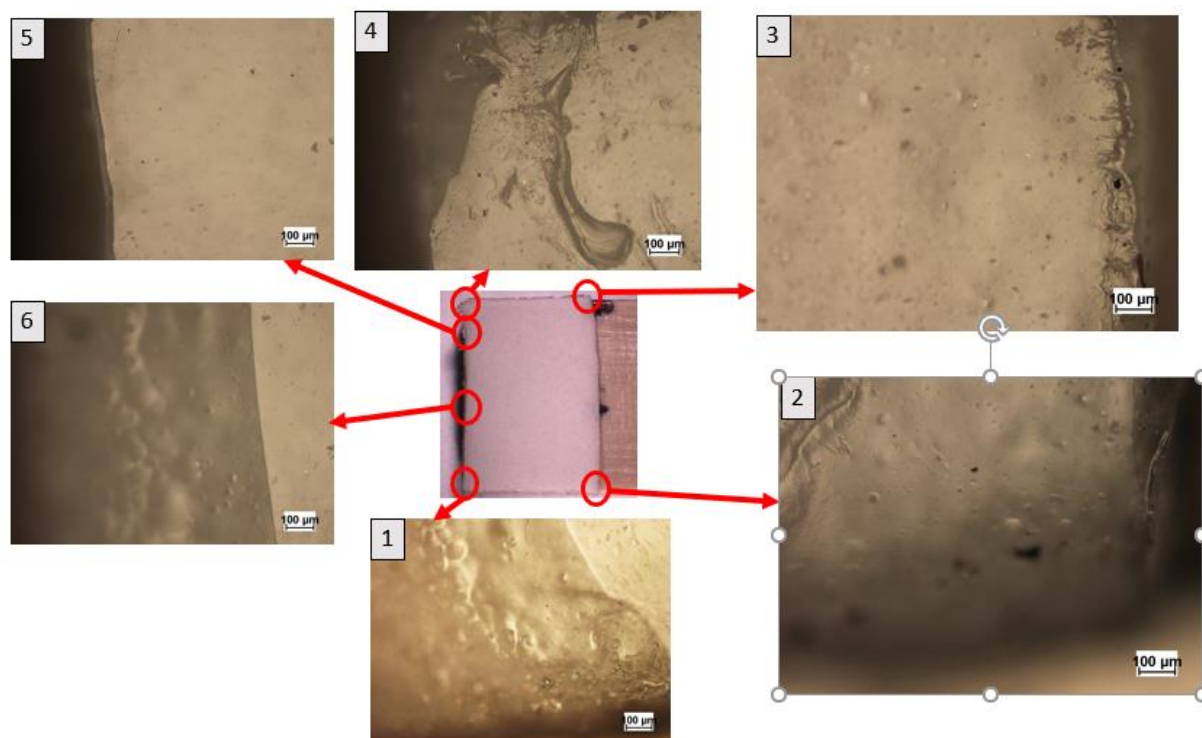
Figure 5.8. Microscopy of Failure Mode A – adhesive failure: surface view.

(a)   (b)
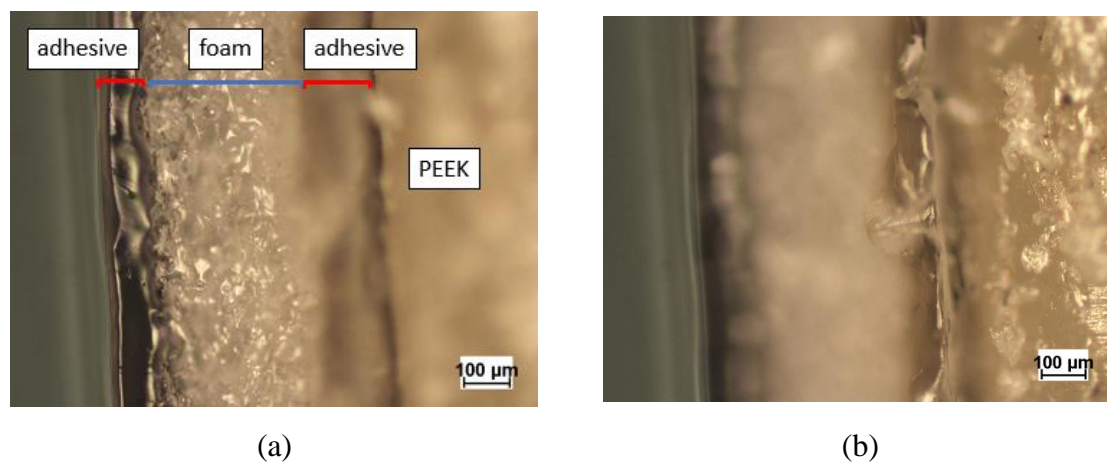
Figure 5.9. Microscopy of Failure Mode A – adhesive failure: section view.

5.6.2. Failure Mode B

For the situation Failure Mode B, in Figure 5.10 it is visible at naked eye that there was adhesion and cohesion failure (of the adhesive layer and foam), the microstructure is very similar to Failure Mode A, but the affected area is larger. The transition between a

darker area where the adhesive coating has been removed (and stuck to the PEM surface) and a lighter area where that adhesive coating still covers the foam is pictured in Figure 5.10.
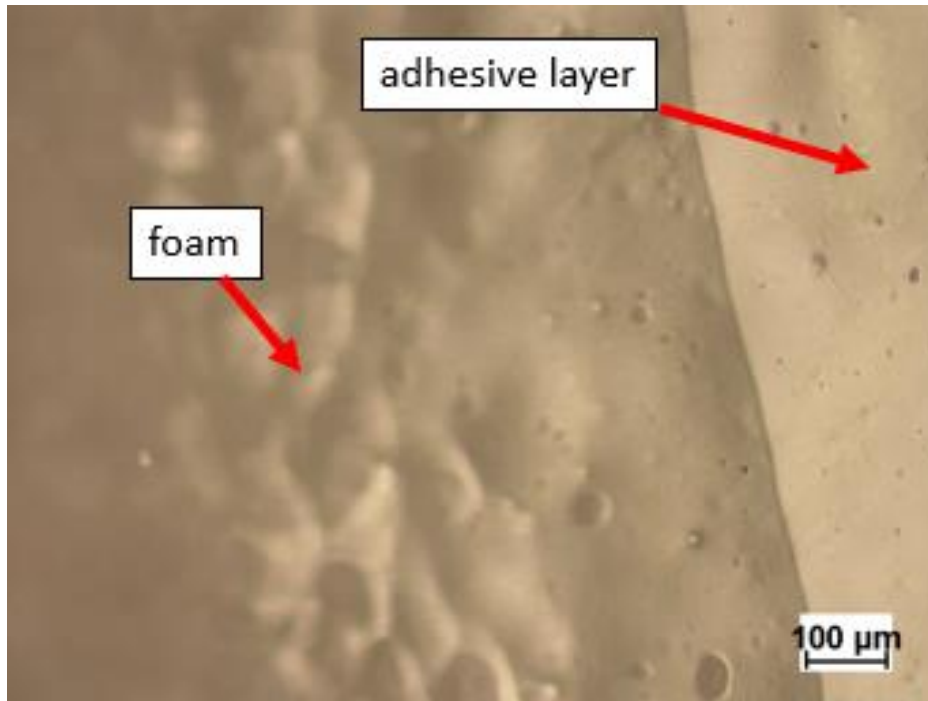
Figure 5.10. Transition between foam and the foam covered in adhesive film.

5.6.3. Failure Mode C

Microscopic observation of the specimens with Failure Mode C was very complicated because, in the points of interest, the depth variation was so high that made it impossible to understand whether adhesive film or foam was being observed. A higher magnification macrograph is shown in Figure 5.11.The image enables to observe that the PSA tape was strained along the edges and in the corners, leading to localized failure by adhesion, in both the adhesive-PEM and adhesive-PEEK interfaces, as well as cohesive failure, in the adhesive and foam backing. This straining pattern shows a round area, within the overlap area, inside which the bond is strong.
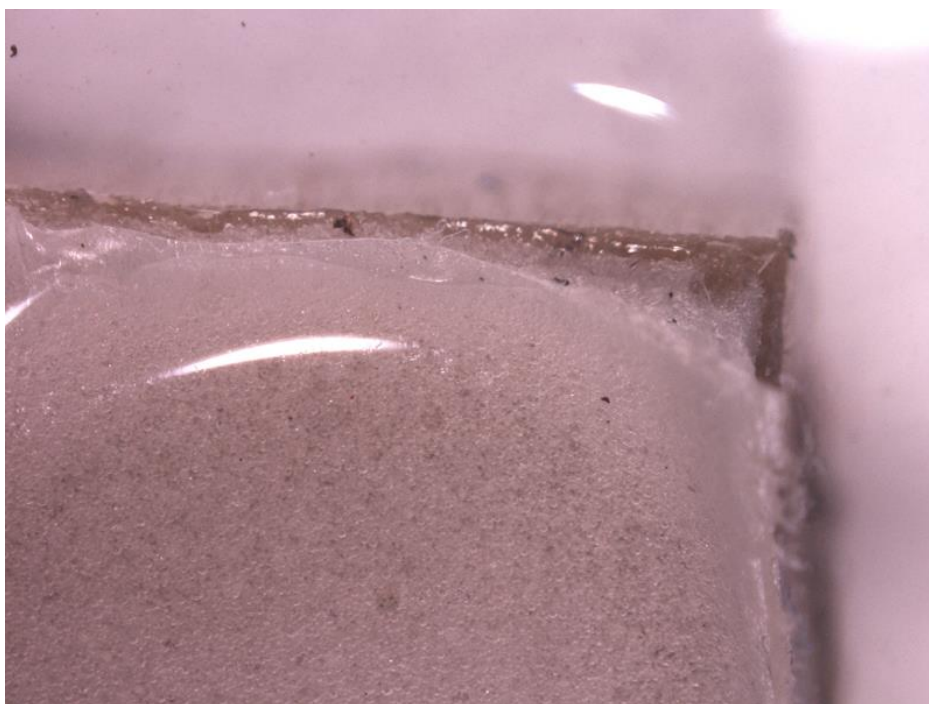
Figure 5.11. Macroscopic detail of corner from joint where there was PEM failure: shiny surface is PEM, adhesive is white foamy matt structure behind, PEEK is the brownish surface at the furthest depth.

6. CONCLUSIONS AND FUTURE WORK

Throughout this work, different adhesive bonded single-lap joints were analysed. These joints involved two different polymers, both with rather low surface energies, which makes its adhesive bonding difficult. Upon finding a viable adhesive, the mechanical performance of the joints was assessed, and a forensic analysis was conducted to understand the failure mechanisms under loading.

It was possible to verify that Nafion is a polymeric film that needs to be machined precisely to avoid the formation of defects conducting to stress concentration and premature failure upon loading. The high demand in terms of cutting precision can be a limitation in the manufacturing of components made from this polymer, since the tooling required may be not always available or cost effective.

Only the pressure sensitive adhesive (PSA) tape was found to be able to establish a strong adhesive bond with such a chemically stable surface like a Nafion PEM, without expensive, complex surface preparation. The other cyanoacrylate, and two other acrylic adhesives (one toughened and another a modified acrylic) did not achieve successful bonds. The same chemical mechanisms that make it possible that only protons can cross the PEM are, most likely, the same that inhibit bonding mechanisms. Although the struggle on the PEEK side was not as demanding, it is worth noting that this polymer is also difficult to bond.

It was possible to confirm that an adequate clamping pressure during the cure of the adhesive joints is necessary. Too little pressure and the intimate contact between the adherends and the adhesive is not enough and too much pressure, suppresses the materials' ability to flow and wet the adherends surface and affects negatively the curing process.

Through the study of the overlap length variation on the mechanical performance of the joint, it was not possible to establish a rigorous relation between the overlap length and the lap-shear strength. However the results showed that a 6 mm overlap length, for the given overlap area and cross-section could already handle a typical PEM fuel cell internal pressure.

It was also possible to understand how the joints failed through a forensic analysis of the joint specimens. The optical microscopies shed a light on the possibility of having either PEM fracture, probably related to defects in the adherend material, which have already been discussed, or a mixture of loss of adhesion and cohesion of the adhesive structure and the adherends. It was possible to find visual evidence that the loss of cohesion and adhesion are localized nearby areas where the stresses are theoretically higher. It would be interesting to face the technical difficulties of using SEM with two electrically insulating polymers to complement this forensic analysis of the adhesive bonded joints.

To improve the system and tackle the manufacturing issues related to assembly and sealing of PEM fuel cells, it would be interesting to test other less conventional PEM materials that could keep up the electrochemical performance of the Nafion, while being more processable. An example of this material would sulphonated PEEK.

If the material cannot be changed due to electrochemical performance and durability, it would be interesting to try new processing techniques. Ultrasonic welding has a major limitation regarding the need for a continuous weld to close the joint all around the frame. Friction stir welding presents a challenge because of heat processability of Nafion.

Another proposal in the follow-up of this work would be environmental testing of the joints in service conditions to assess the durability.

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ANNEX A

Technical Data Sheet

ⓧ RÖCHLING

SUSTAPEEK

Product characteristics

- Excellent dimensional stability
- Flame retardant and self-extinguishing
- Very low smoke density

Product applications

- Medical engineering
- Aviation industry
- Electrical industry

	Test method	Unit	Guideline Value
General properties			
Density	DIN EN ISO 1183-1	g / cm³	1,31
Water absorption	DIN EN ISO 62	%	0,2
Flammability (Thickness 3 mm / 6 mm)	UL 94		V0 / V0
Thermal properties			
Melting temperature	ISO 11357-3	°C	343
Thermal conductivity	DIN 52612-1	W / (m * K)	0,25
Thermal capacity	DIN 52612	kJ / (kg * K)	1,34
Coefficient of linear thermal expansion	DIN 53752	10^{-6} / K	50
Service temperature, long term	Average	°C	-60 ... 250
Service temperature, short term (max.)	Average	°C	310
Heat deflection temperature	DIN EN ISO 75, Verf. A, HDT	°C	152
Electrical properties			
Dielectric constant	IEC 60250		3,2
Dielectric dissipation factor (50 Hz)	IEC 60250		0,001
Volume resistivity	DIN EN 62631-3-1	Ω * cm	$4,9 * 10^{16}$
Surface resistivity	DIN EN 62631-3-2	Ω	10^{16}
Dielectric strength	IEC 60243	kV / mm	20
Mechanical properties			
Yield stress	DIN EN ISO 527	MPa	110
Elongation at break	DIN EN ISO 527	%	20
Tensile modulus of elasticity	DIN EN ISO 527	MPa	4000
Shore hardness	DIN EN ISO 868	scale D	88

The short term maximum application temperature only applies to very low mechanical stress for a few hours. The long term maximum application temperature is based on the thermal ageing of plastics by oxidation, resulting in a decrease of the mechanical properties. This applies to an exposure to temperatures for at least 5.000 hours causing a 50% loss of the tensile strength from the original value (measured at room temperature). This value says nothing about the mechanical strength of the material at high application temperatures. In case of thick-walled parts, only the surface layer is affected by oxidation from high temperatures. With the addition of antioxidants, a better protection of the surface layer is achieved. In any case, the center area of the material remains unaffected. The minimum application temperature is basically influenced by positive stress factors like impact and/or shock under application. The values stated refer to an minimum degree of impact stress. The electrical properties, as stated result from measurements on natural, dry material. With other colours (in particular black) or saturated material, there may be deer differences in the electrical properties. The data stated above are average values ascertained by statistical tests on a regular basis. They are in accordance with DIN EN 15860. They serve as information about our products and are presented as a guide to choose from our range of materials. This, however, does not include an assurance of specific properties or the suitability for particular application purposes that are legally binding. Since the properties also depend on the dimension of the semi-finished products and the degree of crystallisation (e.g. nucleating by pigments), the actual values of the properties of a particular product may differ from the indicated values.

ANNEX B

Loctite 406 datasheet

LOCTITE

Technical Data Sheet

LOCTITE® 406™

(TDS for new formulation of Loctite® 406™) February 2012

PRODUCT DESCRIPTION

LOCTITE® 406™ provides the following product characteristics:

Technology	Cyanoacrylate
Chemical Type	Ethyl cyanoacrylate
Appearance (uncured)	Transparent, colorless to straw colored liquid^{LMS}
Components	One part - requires no mixing
Viscosity	Low
Cure	Humidity
Application	Bonding
Key Substrates	Plastics and Rubbers

This Technical Data Sheet is valid for LOCTITE® 406™ manufactured from the dates outlined in the "Manufacturing Date Reference" section.

LOCTITE® 406™ is designed for bonding of plastics and elastomeric materials where very fast fixturing is required.

Commercial Item Description A-A-3097:
LOCTITE® 406™ has been qualified to Commercial Item Description A-A-3097. **Note:** This is a regional approval. Please contact your local Technical Service Center for more information and clarification.

TYPICAL PROPERTIES OF UNCURED MATERIAL

Specific Gravity @ 25 °C	1.1
Viscosity, Cone & Plate, mPa·s (cP):	
Temperature: 25 °C, Shear Rate: 3,000 s⁻¹	12 to 22^{LMS}
Viscosity, Brookfield - LVF, 25 °C, mPa·s (cP):	
Spindle 1, speed 30 rpm	15 to 25
Flash Point - See SDS	

TYPICAL CURING PERFORMANCE

Under normal conditions, the atmospheric moisture initiates the curing process. Although full functional strength is developed in a relatively short time, curing continues for at least 24 hours before full chemical/solvent resistance is developed.

Cure Speed vs. Substrate
The rate of cure will depend on the substrate used. The table below shows the fixture time achieved on different materials at 22 °C / 50 % relative humidity. This is defined as the time to develop a shear strength of 0.1 N/mm² .

Fixture Time, seconds:

Steel (degreased)	20 to 45
Aluminum (etched)	<5
Neoprene	<5
Rubber, nitrile	<5
ABS	<5
PVC	<5
Polycarbonate	10 to 20
Phenolic	<5

Cure Speed vs. Bond Gap
The rate of cure will depend on the bondline gap. Thin bond lines result in high cure speeds, increasing the bond gap will decrease the rate of cure.

Cure Speed vs. Humidity
The rate of cure will depend on the ambient relative humidity. The best results are achieved when the relative humidity in the working environment is 40% to 60% at 22°C. Lower humidity leads to slower cure. Higher humidity accelerates it, but may impair the final strength of the bond.

Cure Speed vs. Activator
Where cure speed is unacceptably long due to large gaps, applying activator to the surface will improve cure speed. However, this can reduce ultimate strength of the bond and therefore testing is recommended to confirm effect.

TYPICAL PERFORMANCE OF CURED MATERIAL
Adhesive Properties

After 24 hours @ 22 °C
Lap Shear Strength, ISO 4587:

Steel (grit blasted)	N/mm²	15.5
	(psi)	(2,250)
Aluminum (etched)	N/mm²	12
	(psi)	(1,740)
Zinc dichromate	N/mm²	14
	(psi)	(2,030)
ABS	* N/mm²	8.8
	* (psi)	(1,280)
PVC	* N/mm²	8.7
	* (psi)	(1,260)
Polycarbonate	* N/mm²	9.1
	* (psi)	(1,320)
Phenolic	* N/mm²	11.3
	* (psi)	(1,640)
Neoprene	* N/mm²	1
	* (psi)	(150)
Nitrile	* N/mm²	1.2
	* (psi)	()

Henkel

TDS LOCTITE® 406™, February 2012

Block Shear Strength, ISO 13445:

Polycarbonate	N/mm²	13.1
	(psi)	(1,900)
ABS	* N/mm²	23.7
	* (psi)	(3,440)
PVC	N/mm²	1.8
	(psi)	(260)
Phenolic	* N/mm²	13.8
	* (psi)	(2,000)

* substrate failure

Tensile Strength, ISO 6922:

Buna-N	N/mm²	13
	(psi)	(1,890)

After 10 seconds @ 22 °C
Tensile Strength, ISO 6922:

Buna-N	N/mm²	≥6.9[AR]
	(psi)	(≥1,000)

TYPICAL ENVIRONMENTAL RESISTANCE
Cured for 1 week @ 22 °C
Lap Shear Strength, ISO 4587:
Mild Steel (grit blasted)

Hot Strength
Tested at temperature
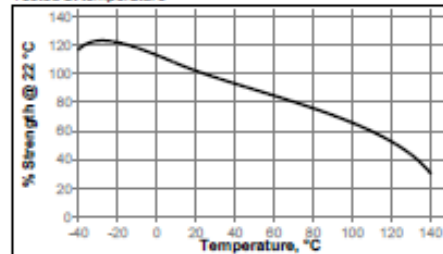
Heat Aging
Aged at temperature indicated and tested @ 22 °C
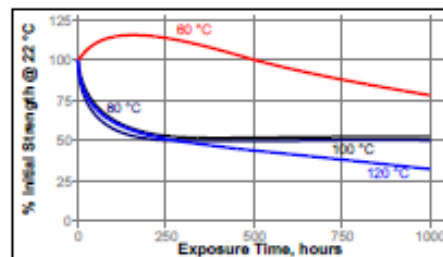
Chemical/Solvent Resistance
Aged under conditions indicated and tested @ 22 °C.

		% of initial strength		
Environment	°C	100 h	500 h	1000 h
Motor oil	40	100	85	70
Gasoline	22	90	100	95
Water	22	55	70	70
Water/glycol	22	85	75	80
Ethanol	22	105	105	100
Isopropanol	22	120	110	120
98% RH	40	50	60	45

Chemical/Solvent Resistance
Aged under conditions indicated and tested @ 22°C.
Lap Shear Strength, ISO 4587, Polycarbonate

		% of initial strength		
Environment	°C	100 h	500 h	1000 h
Air	22	100	100	105
98% RH	40	85	90	85

GENERAL INFORMATION
This product is not recommended for use in pure oxygen and/or oxygen rich systems and should not be selected as a sealant for chlorine or other strong oxidizing materials.

For safe handling information on this product, consult the Safety Data Sheet (SDS).

Directions for use:
1. Bond areas should be clean and free from grease. Clean all surfaces with a Loctite® cleaning solvent and allow to dry.
2. To improve bonding on low energy plastic surfaces, Loctite® Primer may be applied to the bond area. Avoid applying excess Primer. Allow the Primer to dry.
3. LOCTITE® Activator may be used if necessary. Apply it to one bond surface (do not apply activator to the primed surface where Primer is also used). Allow the Activator to dry.
4. Apply adhesive to one of the bond surfaces (do not apply the adhesive to the activated surface). Do not use items like tissue or a brush to spread the adhesive. Assemble the parts within a few seconds. The parts should be accurately located, as the short fixture time leaves little opportunity for adjustment.
5. LOCTITE® Activator can be used to cure fillets of product outside the bond area. Spray or drop the activator on the excess product.
6. Bonds should be held fixed or clamped until adhesive has fixtured.
7. Product should be allowed to develop full strength before subjecting to any service loads (typically 24 to 72 hours after assembly, depending on bond gap, materials and ambient conditions).

Permabond TA4610 datasheet

Permabond®
Engineering Adhesives

PERMABOND® TA4610
Toughened Acrylic Adhesive
Provisional Technical Datasheet

Features & Benefits

- Adhesion to a wide variety of substrates
- Full cure at room temperature
- Bonds polyolefins
- No primer required
- Good impact strength
- Good chemical resistance

Description

PERMABOND® TA4610 is a 2-part, 1:1 toughened acrylic adhesive. It has been developed to bond low surface energy plastics such as polypropylene and polyethylene – with no primer or surface treatment required. It can also be used to bond a wide variety of other materials such as metals and composite materials and is ideal for bonding dissimilar surfaces.

Physical Properties of Uncured Adhesive

	TA4610 A	TA4610 B
Chemical composition	Methacrylate	Methacrylate
Colour	Off-white	Almost colourless
Mixed colour	Off-white	
Viscosity @ 25°C	200,000-500,000 mPa.s (cP) Thixo paste	20,000-30,000 mPa.s (cP)
Specific gravity	1.0	1.0

## Typical Curing Properties

Ratio of use	1 : 1
Maximum gap fill (NB: Contains microparticles to control gap)	1 mm (0.04 in)
Pot life (2g+2g) @23°C	5-8 minutes
Fixture time (0.1 N/mm² shear strength is achieved) @23°C	12-15 minutes
Handling time (0.3 N/mm² shear strength is achieved) @23°C	40-50 minutes
Working strength @23°C	6-8 hours
Full cure @23°C	36-48 hours

Typical Performance of Cured Adhesive

|	
Shear strength (ISO4587)	Polypropylene: 5-8 N/mm² *(725-1200 psi)** Polyethylene: 3-6 N/mm² *(435-870 psi)** Aluminium / PE: 3-6 N/mm² *(435-870 psi)* PTFE: 1.5 N/mm² *(220 psi)* ABS: >4 N/mm² *(>580 psi)** Polycarbonate: >5 N/mm² *(>725 psi)** PVC: >5 N/mm² *(>725 psi)** EPDM: 3-5 N/mm² *(435-725 psi)** PA6: 3-5 N/mm² *(435-725 psi)*

*Substrate failure was observed.

Strength Development
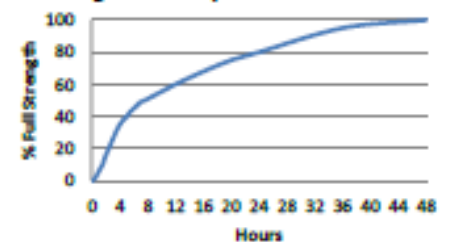
Graph shows typical strength development of bonded components at 23°C. An increase of 8°C in temperature will halve the cure time. Lower temperatures will result in a slower cure time.

Hot Strength
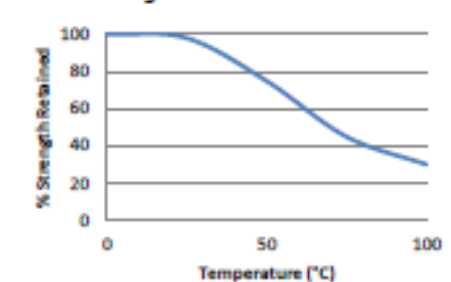
Fully cured lap shear specimens conditioned to pull temperature for 30 minutes before testing at temperature.

TA4610 can withstand higher temperatures for brief periods (such as for paint baking and wave soldering processes) providing the joint is not unduly stressed. The minimum temperature the cured adhesive can be exposed to is -55°C depending on the materials being bonded.

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Additional Information

This product is not recommended for use in contact with strong oxidizing materials. This product may affect some thermoplastics and users must check compatibility of the product with such substrates.

Information regarding the safe handling of this material may be obtained from the safety data sheet (SDS).

Users are reminded that all materials, whether innocuous or not, should be handled in accordance with the principles of good industrial hygiene.

This Technical Datasheet (TDS) offers guideline information and does not constitute a specification.

Surface Preparation

Surfaces should be clean, dry and grease-free before applying the adhesive. Polyolefin surfaces may have traces of mold release agent present – wipe with isopropanol (IPA) solvent and allow to fully evaporate before bonding. If bonding to metal: some metals such as aluminium, copper and its alloys will benefit from light abrasion with emery cloth (or similar) to remove the oxide layer.

Directions for Use

1) Surfaces must be clean, dry and grease-free prior to bonding.
2) Apply a thin bead of adhesive pre-mixed through a static mixer nozzle.
3) Assemble components and clamp.
4) Maintain pressure until handling strength is achieved. The time required will vary according to the joint design and surfaces being bonded.
5) Allow 36-48 hours for adhesive to fully cure.

NB: Adhesive outside of a closed joint (i.e. excess material) will cure more slowly and may feel soft due to air contact. Adhesive inside the joint will cure solid.

Video Links

Surface preparation:
https://youtu.be/8CMOMP7hXjU

Structural acrylic directions for use:
https://youtu.be/YVeKBCVVhYo

Other Products Available

Anaerobics
- Thread lockers - Thread sealants
- Gasket makers - Sealants / retainers

Cyanoacrylates
- Instant adhesives
- For rapid bonding of metals, plastics, rubber and many other materials

Epoxies
- Two-part room temperature cure adhesives
  - Single-part heat cure adhesives
- Modified Technology (MT) flexible grades available

MS-Polymers
- Single-part, moisture-curing, flexible sealants

Polyurethanes
- Two-part room temperature curing adhesives

Toughened Acrylics
- Rapid curing, high strength structural adhesives

UV Light Cured Adhesives
- Glass / plastic bonding
  - Optically clear
  - Non-yellowing

Storage & Handling

Storage Temperature	2 to 7°C (35 to 45°F)

This product may separate slightly – in this instance, invert container to re-dispense.

3M Scotch-Weld DP8010 Blue datasheet

3M
Scotch-Weld™ Structural Plastic Adhesives
DP8010 Blue • DP8010NS Blue

Technical Data Sheet   **October 2018**

Product Description

3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue and DP8010NS Blue are two-part, acrylic-based adhesives (10:1 ratio by volume) that can bond many low surface energy plastics, including many grades of Polypropylene, Polyethylene and TPO's *without special surface preparation.*

These adhesives can replace screws, rivets, plastic welding, and two-step processes which include chemical etchants, priming or surface treatments in many applications.

Features

- Ability to structurally bond polyolefins without special surface preparation
- Regular and Non-Sag Formulations
- Excellent water and humidity resistance
- One step process; no pre-treatment of polyolefin substrates necessary
- Convenient hand-held applicator

- Ability to bond dissimilar Substrates
- Room temperature cure
- Very good chemical resistance
- Solvent-free adhesive system
- Available in bulk

Typical Uncured Properties

Note: The following technical information and data should be considered representative or typical only and should not be used for specification purposes. Unless otherwise indicated, all properties measured at 72°F (22°C).

Property		3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010NS Blue
Color	Base (B)	Blue-Green	
	Accelerator (A)	Clear and nearly colorless	
Viscosity[1]	Base (B)	27,000 cP	64,000 cP
	Accelerator (A)	17,000 – 40,000 cP	17,000 – 40,000 cP
Density	Base (B)	8.5 lb/gal	
	Accelerator (A)	8.3 – 8.7 lb/gal	
Mix ratio	By volume	10:1	
	By weight	10:1	
Work life[2]		Approx. 8 minutes	
Open time[3]		10 minutes	
Skin Time[4]		Approximately 3 minutes (See Below)	
Time to handling strength[5]		60 minutes	

1. Viscosity measured using Brookfield RTV, spindle #7, 20 RPM @ 80°F (27°C)
2. Maximum time that adhesive can remain in a static mixing nozzle and still be expelled without undue force on the applicator.
3. Maximum time allowed after applying adhesive to one substrate before bond must be closed and fixed in place.
4. An open bead line will show some skinning in approximately 3 minutes. It is possible to bond parts with good strength if the parts are made within 10 minutes. Therefore, the adhesive has a 10 minute open time for making bonds.
5. Minimum time required to achieve 50 psi of overlap shear strength, measured on HDPE.

Note: The data in this sheet were generated using the 3M™ EPX Applicator System equipped with an EPX static mixer, according to manufacturer's directions. Thorough hand-mixing will afford comparable results.

3M™ Scotch-Weld™ Structural Plastic Adhesive
DP8010 Blue • DP8010NS Blue

Note: The following technical information and data should be considered representative or typical only and should not be used for specification purposes. Unless otherwise indicated, all properties measured at 72°F (22°C)

Typical Mixed Properties

Property	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue
Color	Blue-Green	
Full cure time	24 hours	
Dispense Viscosity (73°F)	25,000 cP	64,000 cP

Typical Cured Physical Properties

Property	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue
Physical Shore D Hardness Storage Modulus (DMA) Tensile Strength (ASTM D638) Tensile Modulus (ASTM D638) Strain at Break (ASTM D638)	55-60 970 MPa 1300 PSI 77,000 PSI 90%
Thermal Tg (Glass Transition Temperature) (DMA) Coefficient of Thermal Expansion (in/in/°C) — Below Tg / Above Tg	61°C 116 245
Electrical Dielectric Strength (ASTM D 149) Volume Resistivity ((ASTM D 257) Surface Resistivity (ASTM D257) Dielectric Constant (ASTM D150) Dissipation Factor (ASTM D150)	603 V/mil 4.10E+11 (Ω-cm) 8.00E+10 (Ω) 4.36 at 1 KHz 0.068 at 1 KHz

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3M™ Scotch-Weld™ Structural Plastic Adhesive
DP8010 Blue • DP8010NS Blue

Typical Cured Physcial Properties (continued)

Note: The following technical information and data should be considered representative or typical only and should not be used for specification

Overlap Shear (psi)[7], ASTM D1002

Substrate	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010NS Blue
Aluminum (MEK/abrade/MEK)	1960 CF	1780 CF
Cold-rolled steel (MEK/abrade/MEK)	1800 CF	1870 CF
Stainless Steel (MEK/abrade/MEK)	1820 CF	1990 CF
Copper (MEK/abrade/MEK)	1870 CF	1500 CF
Galvanized steel (MEK/abrade/MEK)	1330 CF	840 mixed
PP (IPA wipe)	1150 SF	1150 SF
LDPE (IPA wipe)	360 SF	360 SF
HDPE (IPA wipe)	1040 SF	1100 SF
UHMW-PE (IPA wipe)	770 CF	750 SF
Gelcoat (fiberglass--smooth side)	900 SF	1100 SF
Acrylic	1100 SF	1190 SF
PVC	1730 SF	1740 SF
PC	760 AF	740 AF
ABS	1250 SF	1240 SF
Polystyrene (HIPS)	580 SF	570 SF
FRP (Epoxy)	2830 CF	2860 CF
Acetal	90 AF	70 AF
SMC (Fiberglass--rough side)	760 SF	800 SF
Glass	530 SF	670 SF
PTFE (IPA/abrade/IPA)	320 AF	360 AF

Overlap Shear (psi); Etched Aluminum, at Temperature[7], ASTM D1002

Temperature	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010NS Blue
-20°F (-29°C)	2000 mixed	2000 mixed
73°F (23°C)	1800 CF	1700 CF
120°F (49°C)	1000 mixed	700 mixed
150°F (66°C)	450 AF	340 AF
180°F (82°C)	300 AF	100 AF

6. Overlap shear values measured using ASTM D1002; adhesives allowed to cure for 7 days at room temperature; ½" overlap; 0.010" bond line thickness; samples pulled at 0.1 in/min for metals and 2 in/min for plastics; all surfaces prepared with light abrasion and solvent clean; substrates used were 1/16" thick aluminum and 1/8" thick plastics; failure modes: AF: adhesive failure CF: cohesive failure SF: substrate failure mixed: AF/CF

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3M™ Scotch-Weld™ Structural Plastic Adhesive
DP8010 Blue • DP8010NS Blue

Typical Cured Physcial Properties (continued)

Note: The following technical information and data should be considered representative or typical only and should not be used for specification

Environmental Resistance[8] Expressed as Percent Retention of Control Strength
(Measured on 1/8" thick HDPE via Overlap Shear, ASTM D1002)

Condition	Substrate	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010NS Blue
Control	HDPE	100% SF	100 % SF
160°F water soak		80% CF	80% CF
150°F/80% RH		95% CF	97% CF
NaOH 10% by wt		100% SF/CF	100% SF
HCl 16% by volume		100% SF	100% SF/CF
IPA soak		95% CF	91% CF
Diesel Fuel soak		97% SF/CF	93% SF
50% Antifreeze soak		100% SF/CF	100% SF
Gasoline soak		70% CF	70% CF
Acetone soak		20% AF	25% AF

8. Values indicate overlap shear test performance retained after 14 days of continuous exposure relative to a control sample left at room temperature; samples conditioned for 7 days at room temperature and 50% relative humidity prior to tests.

Floating Roller Peel (lb/inch width)[9] ASTM D3167

Substrate	3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010 Blue and 3M™ Scotch-Weld™ Structural Plastic Adhesive DP8010NS Blue
HDPE	Substrate Failure

9. Floating roller peel values measured using ASTM D3167; allowed to cure for 24 hours at room temperature; 1" wide samples; 0.017" bond line thickness; samples pulled at 20 in/min. Flexible HDPE was 1mm thick and rigid HDPE was 4.8mm thick.

AF: adhesive failure   CF: cohesive failure SF: substrate failure

Directions For Use

1. To obtain the highest strength structural bonds, paint, oxide films, oils, dust, mold release agents, and all other surface contaminants must be completely removed. The amount of surface preparation depends on the required bond strength and environmental aging resistance desired by user. For suggested surface preparations on common substrates, see the section on surface preparation.

2. Mixing
For Duo-Pak Cartridges
Store cartridges with cap end up to allow any air bubbles to rise towards the tip. To use, simply insert the cartridge into the EPX applicator and start the plunger into the cylinders using light pressure on the trigger. Then remove the cap and expel a small amount of adhesive to ensure material flows freely from both sides of cartridge. For automatic mixing, attach an EPX mixing nozzle to the cartridge and begin dispensing the adhesive. For hand mixing, expel the desired amount of adhesive and mix thoroughly. Mix approximately 15 seconds after obtaining a uniform color.

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3M™ Scotch-Weld™ Structural Plastic Adhesive
DP8010 Blue • DP8010NS Blue

For Bulk Containers

Directions For Use (continued)

Mix thoroughly by weight or volume in the proportion specified on the product label or in the typical uncured properties section. Mix approximately 15 seconds after obtaining a uniform color.

3. Apply adhesive and join surfaces within the open time listed for the specific product. Larger quantities and/or higher temperatures will reduce this working time.
4. Allow adhesive to cure at 60°F (16°C) or above until completely firm. Applying heat up to 150°F (66°C) will increase cure speed.
5. Keep parts from moving during cure. Apply contact pressure or fixture in place if necessary. Optimum bond line thickness ranges from 0.005 to 0.020 inch; shear strength will be maximized with thinner bond lines, while peel strength reaches a maximum with thicker bond lines.
6. Excess uncured adhesive can be cleaned up with ketone type solvents.*

*Note: When using solvents, extinguish all ignition sources, including pilot lights, and follow the manufacturer's precautions and directions for use.

Surface Preparation

3M™ Scotch-Weld™ Structural Plastic Adhesives are designed to be used on metal, wood, and most plastic surfaces. The following cleaning methods are suggested for common surfaces:

Steel:
1. Wipe free of dust and dirt with pure solvent such as acetone or isopropyl alcohol.*
2. Sandblast or abrade using clean fine grit abrasives.
3. Wipe again with clean solvent to remove loose particles.*

Aluminum:
1. Wipe free of dust and dirt with pure solvent such as acetone or isopropyl alcohol.*
2. Sandblast or abrade using clean fine grit abrasives.
3. Wipe again with clean solvent to remove loose particles.*
4. When using a primer, apply adhesive within 4 hours of primer application.

Plastics/Rubbers:
1. Wipe with isopropyl alcohol.*
2. Abrade using fine grit abrasives.
3. Wipe with isopropyl alcohol.*

Glass:
1. Solvent wipe surface using acetone or MEK.*
2. Apply a thin coating of a silane adhesion promoter to the glass surfaces to be bonded and allow to dry completely before bonding.

*Note: When using solvents, extinguish all ignition sources, including pilot lights, and follow the manufacturer's precautions and directions for use.

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3M™ Scotch-Weld™ Structural Plastic Adhesive
DP8010 Blue • DP8010NS Blue

Storage	Store product at 40°F (4°C). Do not freeze. Allow product to reach room temperature prior to use.
Shelf Life	3M™ Scotch-Weld™ Structural Plastic Adhesives when stored in unopened original containers kept at recommended storage conditions have a shelf life of 3 months for 55 gal. drums, 9 months for 5 gal. pails and 18 months in duo-pak
Precautionary Information	Refer to Product Label and Material Safety Data Sheet for health and safety information before using this product. For additional health and safety information, call 1-800-364-3577 or 651-737-6501.
For Additional Information	To request additional product information or to arrange for sales assistance, call toll free 1-800-362-3550 or visit www.3M.com/structuraladhesives.
Technical Information	The technical information, guidance, and other statements contained in this document or otherwise provided by 3M are based upon records, tests, or experience that 3M believes to be reliable, but the accuracy, completeness, and representative nature of such information is not guaranteed. Such information is intended for people with knowledge and technical skills sufficient to assess and apply their own informed judgment to the information. No license under any 3M or third party intellectual property rights is granted or implied with this information.
Product Selection and Use	Many factors beyond 3M's control and uniquely within user's knowledge and control can affect the use and performance of a 3M product in a particular application. As a result, customer is solely responsible for evaluating the product and determining whether it is appropriate and suitable for customer's application, including conducting a workplace hazard assessment and reviewing all applicable regulations and standards (e.g., OSHA, ANSI, etc.). Failure to properly evaluate, select, and use a 3M product and appropriate safety products, or to meet all applicable safety regulations, may result in injury, sickness, death, and/or harm to property.
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This Industrial Adhesives and Tapes Division product was manufactured under a 3M quality system registered to ISO 9001 standards.

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3M VHB 5952

**3M** Science.
Applied to Life.™

ASK A 3M EXPERT

April, 2019

3M™ VHB™ Tape 5952

Product Description

Finite Element Analysis (FEA) data is available for this product at: 3m.com/FEA

3M™ VHB™ Tape 5952 is a 0.045 inch (1.1 mm) thick black double coated acrylic foam tape with PE film liner. The modified acrylic adhesive on both sides bonds to a broad range of high, medium and medium/low surface energy substrates including metals, glass and a wide variety of plastics and paints, including many powder coated paints. The very conformable foam provides good contact between substrates even when they are slightly mismatched. 3M™ VHB™ Tape 5952 is part of the 5952 tape family. Each product in this family has modified acrylic adhesive and very conformable foam but varies in thickness, color and liner type.
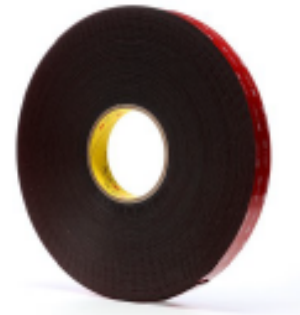
Product Features

* Fast and easy-to-use permanent bonding method provides high strength and long-term durability
* Virtually invisible fastening keeps surfaces smooth
* Can replace mechanical fasteners (rivets, welding, screws) or liquid adhesives
* Black, 0.045 in (1.1 mm), modified acrylic adhesive and very conformable acrylic foam core bonds to a wide variety of substrates including powder coated paints and irregular surfaces
* Eliminate drilling, grinding, refinishing, screwing, welding and clean-up
* Creates a permanent seal against water, moisture and more by offering better gap filling capabilities
* Pressure sensitive adhesive bonds on contact to provide immediate handling strength
* Allows the use of thinner, lighter weight and dissimilar materials

3M™ VHB™ Tape 5952

Technical Information Note

The following technical information and data should be considered representative or typical only and should not be used for specification purposes.

Typical Physical Properties

Property	Values	
Color	Black	
Tape Thickness	1.1 mm	0.045 in
Thickness Tolerance	±10 %	
Adhesive Type	Modified Acrylic	
Foam Type	Very Conformable Acrylic Foam	
Density	590 kg/m³	37 lb/ft³
Liner	PE Film	
Liner Thickness	0.13 mm	0.005 in
Liner Color	Red (printed)	

Typical Performance Characteristics

Property	Values		Method	Dwell/Cure Time	Test Condition	Substrate	Notes
90° Peel Adhesion	39 N/cm	22 lb/in	ASTM D3330	72 hr @ Room Temperature	Room Temperature	Stainless Steel	Jaw speed 12 in/min (304.8 mm/min). Average force to remove is measured.
Normal Tensile	480 kPa	70 lb/in²	ASTM D897	72 hr @ Room Temperature	Room Temperature	Aluminum	1 in² (6.45 cm²), Jaw Speed 2 in/min (50.8 mm/min). Peak force to separate is measured.
Dynamic Overlap Shear	550 kPa	80 lb/in²	ASTM D1002	72 hr @ Room Temperature	Room Temperature	Stainless Steel	1 in² (6.45 cm²), Jaw speed 0.5 in/min (12.7 mm/min). Peak force to separate is measured.
Short Term Temperature Tolerance	149 °C	300 °F					No change in room temperature dynamic shear properties following 4 hours conditioning at indicated temperature with 100 g/static load. (Represents minutes, hours in a process type temperature exposure).
Long Term Temperature Tolerance	93 °C	200 °F					Maximum temperature where tape supports at least 250 g load per 0.5 in² in static shear for 10,000 minutes. (Represents continuous exposure for days or weeks).

3M™ VHB™ Tape 5952

Typical Performance Characteristics (continued)

Static Shear	Test Condition
1000 g	Room Temperature
500 g	66°C (150°F)
250 g	93°C (200°F)

Property: Static Shear
Method: ASTM D3654
Substrate: Stainless Steel
notes: Tested at various temperatures and gram loadings. 0.5 in² (3.23 cm²). Will hold listed weight for 10,000 minutes (approximately 7 days).

Available Sizes

Property	Values	
Standard Length	32.9 m	36 yd
Minimum Available Width	6.4 mm	0.25 in
Maximum Available Width	1219 mm	48 in
Normal Slitting Tolerance	±0.79 mm	±1/32 in
Core Size (ID)	76.2 mm	3 in

Available Sizes:

Available Sizes				Maximum Roll Length		
Tape Thickness inches (mm)	Standard Length yards (meters)	Minimum Width inches (mm)	Maximum Width inches (mm)	Width 1/2 up to 1/2" (6.4mm up to 12mm) yards (meters)	Width >1/2" up to 1/2" (>6.4mm up to 41.7mm) yards (meters)	Width 1/2" and wider (12.7mm and wider) yards (meters)
<0.015 (0.4)	72 (65.5)	9.5 (0.25)	46 (1168)	364 N/A	N/A N/A	See Note Below
0.015/0.015 (0.4)	72 (65.5)	0.25 (6)	45 (1250)	144 (131.7)	175 (160.0)	360 (329.2)
0.025 (0.6)	72 (65.5)	0.25 (6)	45 (1156)	72 (65.3)	108 (98.8)	115 (105.0)
0.052 (0.8)	72 (65.5)	0.25 (6)	46 (1270)	72 (65.3)	108 (98.8)	115 (105.0)
0.040 (1.0)	36 (32.9)	0.25 (6)	46 (1270)	72 (65.3)	108 (98.8)	144 (131.7)
0.045 (1.1)	36 (32.9)	0.25 (6)	46 (1270)	72 (65.3)	108 (98.8)	144 (131.7)
0.062 (1.5)	36 (32.9)	0.25 (6)	46 (1168)	72 (65.3)	72 (65.8)	108 (95.8)
0.090 (2.3)	36 (32.9)	0.25 (6)	46 (1168)	36 (32.9)	36 (32.9)	33 (65.3)

*Exception – 5952 (P) max. width 46 inches (1168 mm), 5925 (P) max. width 47 inches (1194 mm).

Note: 5952 family tapes thinner than 0.015 in (0.4 mm) have max. length 360 yd (329.2 m) for widths 1 in (25 mm) to 8 in (205 mm) and 180 yd (164.6 m) for all other widths.

Converted Parts

In addition to standard and custom roll sizes available from 3M through the distribution network, 3M™ VHB™ Tapes are also available in limitless shapes and sizes through the 3M Converter network. For additional information, contact 3M Converter Markets at 1-800-223-7427 or on the web at www.3M.com/converter.
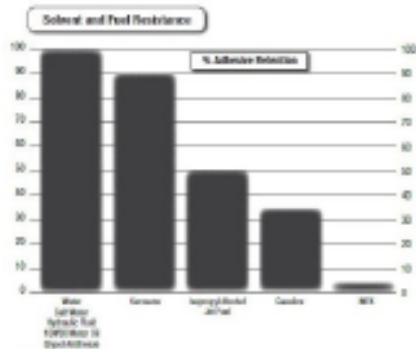
3M™ VHB™ Tape 5952

UL746C Listings

Solvent and Fuel Resistance

3M™ VHB™ Tape 5952

Additional Typical Performance Characteristics

Property	Values		Method	Test Condition
Water Vapor Transmission Rate	37.1 g/m²/24 hr		ASTM F1249	@ 38°C/100% RH
Shear Modulus	3 × 10^5 Pa			
Poisson's Ratio	0.35			
Coefficient of Thermal Expansion	180 × 10^-6 m/m/°C	100 × 10^-6 In/In/°F		

Electrical and Thermal Properties

Property	Values		Method	Test Condition
Dielectric Constant	2.14		ASTM D150	1 KHz, Room Temperature
Dielectric Constant	1.95		ASTM D150	1MHz, Room Temperature
Dissipation Factor	0.0065		ASTM D150	1 KHz, Room Temperature
Dissipation Factor	0.0506		ASTM D150	1MHz, Room Temperature
Dielectric Strength	18 V/μm	455 V/mil	ASTM D140	
Thermal Conductivity (k value)	0.05 W/m/K	0.4 (btu-ft)/(h-ft²-°F)		
Volume Resistivity	2.5 × 10^14 Ω-cm		ASTM D257	Room Temperature
Surface Resistivity	>10^16 Ω/sq		ASTM D257	Room Temperature

Design Considerations

Adhesion to the substrate is important in achieving bonding success. Adhesives must flow onto the substrate surfaces in order to achieve intimate contact area and allow the molecular force of attraction to develop. The degree of flow of the adhesive on the substrate is largely determined by the surface energy of the substrate. 3M™ VHB™ 5952 family tapes bond well to high (HSE), medium (MSE), and medium/low (M/LSE) surface energy materials. The image below shows typical materials in these categories.

Achieving good contact is also important. The necessary thickness of tape depends on the rigidity of substrates and their flatness irregularity. While the 3M™ VHB™ Tapes will conform to a certain amount of irregularity, they will not flow to fill gaps between the materials. For bonding rigid materials with normal flatness, consider use of tapes with thickness of 45 mils (1.1 mm) or greater. As the substrate flexibility increases thinner tapes can be considered.

Using the right amount of tape is important to handle the expected stresses. Because 3M™ VHB™ Tapes are viscoelastic by nature their strength and stiffness is a function of the rate at which they are stressed. They behave stronger with relatively faster rate of stress load (dynamic stresses) and will tend to show creep behavior with stress load acting over a long period of time (static stresses). As a general rule, for static loads, approximately four square inches of tape should be used for each pound (57 cm² of tape per kg) of weight to be supported in order to prevent excessive creep. For dynamic loads a useful design factor is 12 lb/in2 (85 kPa) for most dynamic stresses in general applications.

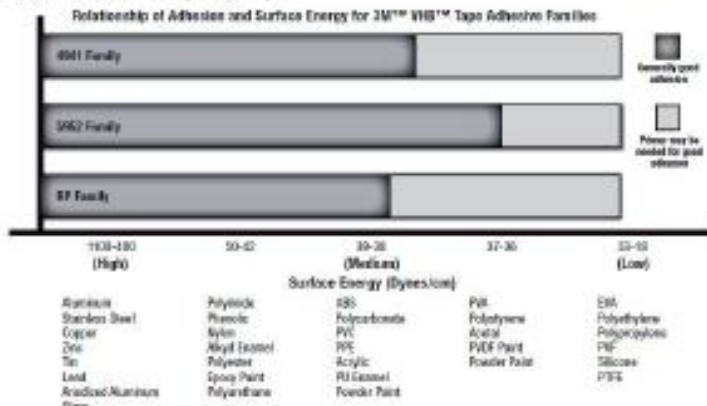Allow for thermal expansion/contraction. 3M™ VHB™ Tapes can perform well in applications where two bonded surfaces may expand and contract differentially. Assuming good adhesion to the substrates, the tapes can typically tolerate differential movement in the shear plane up to 3 times their thickness.

Bond Flexibility: While an advantage for many applications where allowing differential movement is a benefit, the tape bonds are typically more flexible than alternative bonding methods. Suitable design modifications or periodic use of rigid fasteners or adhesives may be needed if additional stiffness is required.

Performance in Severe Cold Temperature can be challenging. Applications which require performance at severe cold temperatures must be thoroughly evaluated by the user if the intended use will subject the tape product to high impact stresses. A technical bulletin "3M™ VHB™ Tape Cold Temperature Performance" (70-0707-3991-0) is available for additional information.

This illustration demonstrates the effect of surface energy on adhesive interfacial contact. High surface energy materials draw the adhesive closer for high bond strength.

Relationship of Adhesion and Surface Energy for 3M™ VHB™ Tape Adhesive Families

	1108-480 (High)	50-42	39-38 (Medium)	37-36	33-93 (Low)

Surface Energy (Dynes/cm)

Aluminum	Polyimide	ABS	PVF	EVA
Stainless Steel	Phenolic	Polycarbonate	Polystyrene	Polyethylene
Copper	Nylon	PVC	Acetal	Polypropylene
Zinc	Alkyd Enamel	PPE	PVDF Paint	PVF
Tin	Polyester	Acrylic	Powder Paint	Silicone
Lead	Epoxy Paint	PU Enamel		PTFE
Anodized Aluminum	Polyurethane	Powder Paint		
Glass				

NOTES: There are a wide variety of formulations, surfaces, finishes and surface treatments available on substrate materials which can affect adhesion. This chart is intended to provide only a rough estimate of the adhesion levels which can be expected on some common materials relative to a reference surface such as aluminum.

Even tape can affect and/or limit maximum adhesive strength.

3M™ VHB™ Tape 5952

Handling/Application Information

Application Techniques

Clean: Most substrates are best prepared by cleaning with a 50:50 mixture of isopropyl alcohol (IPA*) and water prior to applying 3M™ VHB™ Tapes.

Exceptions to the general procedure that may require additional surface preparation include:

• Heavy Oils: A degreaser or solvent-based cleaner may be required to remove heavy oil or grease from a surface and should be followed by cleaning with IPA/water.

• Abrasion: Abrading a surface, followed by cleaning with IPA/water, can remove heavy dirt or oxidation and can increase surface area to improve adhesion.

• Adhesion Promoters: Priming a surface can significantly improve initial and ultimate adhesion to many materials such as plastics and paints.

• Porous surfaces: Most porous and fibered materials such as wood, particleboard, concrete, etc. need to be sealed to provide a unified surface.

• Unique Materials: Special surface preparation may be needed for glass and glass-like materials, copper and copper containing metals, and plastics or rubber that contain components that migrate (e.g. plasticizers).

Refer to 3M Technical Bulletin "Surface Preparation for 3M™ VHB™ Tape Applications" for additional details and suggestions. (70-0704-8701-5)

*Note: These cleaner solutions contain greater than 250 g/l of volatile organic compounds (VOC). Please consult your local Air Quality Regulations to be sure the cleaner is compliant. When using solvents, be sure to follow the manufacturer's precautions and directions for use when handling such materials.

Pressure: Bond strength is dependent upon the amount of adhesive-to-surface contact developed. Firm application pressure develops better adhesive contact and helps improve bond strength. Typically, good surface contact can be attained by applying enough pressure to insure that the tape experiences approximately 15 psi (100 kPa) pressure. Either roller or platen pressure can be used. Note that rigid surfaces may require 2 or 3 times that much pressure to make the tape experience 15 psi.

Temperature: Ideal application temperature range is 70°F to 100°F (21°C to 38°C). Pressure sensitive adhesives use viscous flow to achieve substrate contact area. Minimum suggested application temperature for the 3M™ VHB™ Tape 5952 family is 50°F (10°C). Minimum application temperature does vary by 3M™ VHB™ tape family and ranges from 32°F to 60°F (0°C to 15°C)

Note: Initial tape application to surfaces at temperatures below these suggested minimums is not recommended because the adhesive becomes too firm to adhere readily. However, once properly applied, low temperature holding is generally satisfactory. To obtain good performance with all 3M™ VHB™ Tapes, it is important to ensure that the surfaces are dry and free of condensed moisture.

Time: After application, the bond strength will increase as the adhesive flows onto the surface (also referred to as "wet out"). At room temperature approximately 50% of ultimate bond strength will be achieved after 20 minutes, 90% after 24 hours and 100% after 72 hours. This flow is faster at higher temperatures and slower at lower temperatures. Ultimate bond strength can be achieved more quickly (and in some cases bond strength can be increased) by exposure of the bond to elevated temperatures (e.g. 150°F [66°C] for 1 hour). This can provide better adhesive wetout onto the substrates. Abrasion of the surfaces or the use of primers/ adhesion promoters can also have the effect of increasing bond strength and achieving ultimate bond strength more quickly.

Bond Typical Build vs. Time
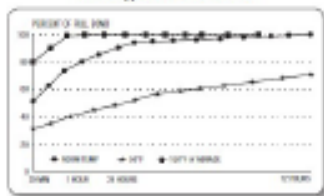
3M™ VHB™ Tape 5952

Storage and Shelf Life

All 3M™ VHB™ Tapes have a shelf life of 24 months from date of manufacture when stored at 40°F to 100°F (4°C to 38°C) and 0-95% relative humidity. The optimum storage conditions are 72°F (22°C) and 50% relative humidity.
Performance of tapes is not projected to change even after shelf life expires; however, 3M does suggest that 3M™ VHB™ Tapes are used prior to the shelf life date whenever possible.
The manufacturing date is available on all 3M™ VHB™ Tapes as the lot number, typically marked on the core or on a label on the outer roll lap. The lot number, typically a 4 digit code, is a Julian date (Y D D D). The first digit refers to the year of manufacture, the last 3 digits refer to the days after January 1. Example: A lot number of 7266 (or 17266) would translate to a date of manufacture of Sept. 23 (266th day of year) in 2017.

Industry Specifications

UL 746C (File MH 17478)
EN 45545 test report for details (ISO 5660-1, ISO 5659-2)

Trademarks

3M and VHB are trademarks of 3M Company.

References

Property	Values
3m.com Product Page	https://www.3m.com/3M/en_US/company-us/all-3m-products/~/3M-VHB-Tape-5952?N=5002385+3293242417&rt=rud
Safety Data Sheet (SDS)	https://www.3m.com/3M/en_US/company-us/SDS-search/results/?gssAction=mdsSRA&mdsLocale=en_US&co=ptn&q=5952

Family Group

	5906	5908	5909	5915	5915P	5915WF	5925	5925P	5925WF	5930	5907
Color	Black	Black	Black	Black	Black	White	Black	Black	White	Black	Black
Tape Thickness (mm)	0.15	0.25	0.3	0.4	0.4	0.4	0.6	0.6	0.6	0.8	0.2
Adhesive Type	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic	Modified Acrylic
Foam Type	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam	Very Conforma ble Acrylic Foam
Liner	PET	PET	PET	PE Film	PCK Paper	PE Film	PE Film	PCK Paper	PE Film	PE Film	PET
Liner Thickness (mm)	0.08	0.08	0.08	0.13	0.1	0.13	0.13	0.1	0.13	0.13	0.08
Liner Color	Clear	Clear	Clear	Red (printed)	White (printed)	Red (printed)	Red (printed)	White (printed)	Red (printed)	Red (printed)	Clear

ISO Statement

This Industrial Adhesives and Tapes Division product was manufactured under a 3M quality system registered to ISO 9001 standards.

3M VHB LSE060/110

3M

VHB™ Tape LSE Series

Product Data Sheet

October 2019
Supersedes: August 2019

Product Description

3M™ VHB™ Tape LSE family are white conformable double coated acrylic foam tapes with a high initial tack and a soft foam.
Its design enables bonding of many low surface energy substrates/materials.
3M VHB LSE white tapes are available in three different thicknesses with a 3M branded red siliconised polyethylene film liner.

Key Features

- Double-coated acrylic foam tape
- 100% closed cell acrylic foam
- Multi material bonding for high, medium or low surface energy substrates including many metals (e.g. stainless steel), composites and plastics (e.g. PP, PA)
- Enables bonding of many LSE substrates without primer
- Good low temperature tack
- Soft foam enables stress relaxation & an easy application
- High initial tack
- For indoor and outdoor applications

Applications & Benefits

- Capability to bond to LSE substrates without primer makes it a good fit for applications in many industries such as plastics processing, transportation, appliances and signage

Physical Properties

		LSE-060WF	LSE-110WF	LSE-160WF
Adhesive & Carrier		Modified Acrylic on Conformable Acrylic Foam (closed cell)		
Thickness acc. to ASTM D-3652		0.60 mm	1.10 mm	1.60 mm
Density		716 kg/m³		
Release Liner		3M branded red siliconised polyethylene film		
Tape Colour		White		

Performance Characteristics Type	LSE-060WF	LSE-110WF	LSE-160WF
90 " Peel adhesion to Stainless Steel acc. to ASTM D3330, 90° peel angle @ RT, after 72h @ RT dwell	30 N/cm	44 N/cm	54 N/cm
90 " Peel adhesion to Polypropylene acc. to ASTM D3330, 90° peel angle @ RT, after 72h @ RT dwell	24 N/cm	42 N/cm	51 N/cm
90 " Peel adhesion to Glass acc. to ASTM D3330, 90° peel angle @ RT, after 72h @ RT dwell	29 N/cm	43 N/cm	51 N/cm
90 " Peel adhesion to ABS acc. to ASTM D3330, 90° peel angle @ RT, after 72h @ RT dwell	24 N/cm	40 N/cm	47 N/cm
Static Shear Strength on Stainless Steel acc. to ASTM D3654, after 72h @ RT dwell (Weight held for 10.000 minutes, 3.22cm² (0.5in²), vertical test direction	23 °C - 1000 g 70 °C – 500 g 90 °C – 250 g		
Static Shear Strength on Polypropylene acc. to ASTM D3654, after 72h @ RT dwell (Weight held for 10.000 minutes, 3.22cm² (0.5in²), vertical test direction Limited by substrate	23 " C - 1000 g 70 °C – 500 g 90 " C - 500 g		
Dynamic Shear acc. to ASTM D1002 on stainless steel, after 72h @ RT dwell	525 N /6.54cm²	382 N /6.54cm²	347 N /6.54cm²
Normal Tensile (T-Block) acc. to ASTM D897 to Aluminium @ RT, after 72h @ RT dwell, test speed 50 mm/min	365 N /6.54cm²	309 N /6.54cm²	290 N /6.54cm²
Temperature Performance	Short term (minutes, hours): 150 °C Long term (days, weeks): 90 °C		

Application Temperature Ideal application temperature range 10 °C to 38 °C. For certain applications and substrates 3M™ VHB ™ LSE can be applied at temperatures as low as 0 °C if the surface is frost free.
Testing on application-specific substrates is recommended to confirm adhesion at temperatures <10°C.
Pressure sensitive adhesives use viscous flow to achieve substrate contact area. To obtain good performance, it is important to ensure that the surfaces are clean, dry and free of condensed moisture.

Shelf Life	18 months from date of production when stored at 16 °C – 25 °C and 40-65 % relative humidity. Performance of tapes is not projected to change even after shelf life expires; however, 3M does suggest that 3M™ VHB™ Tapes are used prior to the shelf life date whenever possible.
Important Notice	All statements, technical information and recommendations contained in this document are based upon tests or experience that 3M believes are reliable. However, many factors beyond 3M's control can affect the use and performance of a 3M product in a particular application, including the conditions under which the product is used and the time and environmental conditions in which the product is expected to perform. Since these factors are uniquely within the user's knowledge and control, it is essential that the user evaluate the 3M product to determine whether it is fit for a particular purpose and suitable for the user's method or application. All questions of liability relating to this product are governed by the terms of the sale subject, where applicable, to the prevailing law.

Values presented have been determined by standard test methods and are average values not to be used for specification purposes. Our recommendations on the use of our products are based on tests believed to be reliable, but we would ask that you conduct your own tests to determine their suitability for your applications. This is because 3M cannot accept any responsibility or liability direct or consequential for loss or damage caused as a result of our recommendations.

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