Czech Technical University in Prague
Faculty of Mechanical Engineering

Bachelor Thesis

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Methods of adhesive bonding joints testing

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Supervisor: **Prof. RNDr. Petr Špatenka, CSc.**

Prague, 2016

Prohlášení autora práce / Statement of author

Prohlašuji, že jsem předloženou práci vypracovala samostatně a že jsem uvedla veškeré použité informační zdroje.

I declare that I carried out this bachelor thesis independently, and only with the cited sources, literature and other professional sources.

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Cíl:

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Postup:

1. Literární rešerše týkající se metod testování spojů.
2. Zhotovit přehled platných norem a porovnat popsané metody.
3. Vybrat metodu vhodnou k aplikaci v laboratořích UMI a provést experiment.
4. Diskuse a závěr.

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[1] Handbook of Adhesives and Sealants (McGraw-Hill Handbooks), Second Edition. Edward M. Petrie. McGraw-Hill Education; 2 edition (January 2, 2007) ISBN-13: 978-0-07-147916-5

[2] Polymerní kompozitní materiály. Gottfried W. Ehrenstein. Springer

[3] CSN a ISO normy - vyhledání je součástí práce

Vedoucí bakalářské práce: Prof. RNDr. Petr Špatenka, CSc.

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Datum zadání bakalářské práce: 25. dubna 2016

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Abstract

Adhesives are means for creating part assemblies with a wide scale of use in industry and therefore a proper knowledge of adhesive properties is necessary. For it is propitious that when a property is tested, results from different test performers are comparable, standards for various testing methods were established. In theoretical part a few of standards that relate to this topic are summarized and compared. In experimental part six adhesives suitable for bonding of polyethylene to aluminium will be tested and compared according to the EN 1465 standard, also influence of plasma surface treatment of polyethylene on adhesive bond strength will be examined.

Abstrakt

Lepidla jsou jedním ze způsobů spojování dílů do sestav. Mají široké využití v průmyslu a proto je nutná řádná znalost vlatsností jdnotlivých lepidel. Protože je potřeba, aby při testování jdnotlivých vlastností byly porovnatelné výsledky prováděné na jiných pracovištích, byly zavedeny normy, které stanovují podmínky testování a příprav vzorků. V této práci budou v teoretické části rozebrány a porovnány některé normy zabývající se danou problematikou. V Experimentální části bude podle normy EN 1465 porovnáno šest lepidel vhodných pro lepení kovu (hliník) s plastem (polyethylen), dále pak porovnání vlivu plazmově upaveného a neupraveného povrhu polyethylenu na pevnost spoje.

Acknowledgements

I would like to express my thanks to Prof. RNDr. Petr Špatenka, CSc. for supervising of this work, advices and hints he gave me and for educating me in academic and non-academic matters through the pleasant conversations we had. As further I would like to thank Ing. Zdeňka Jeníková, Ph.D. for her patience and help with laboratory part of the experiment.
Also I am thankful for adhesive samples provided by 3M Česko, spol. s.r.o. company and to the SurfaceTreat, a.s. company for securing and plasma treatment of polyethylene samples, for provision of space to perform bonding and curing process of experiment and their splendid help, so I can still use both my hand separately.

I am grateful to my family for their patience and support through my studies, for moments of silence when I needed to focus or rest and for moments on adventures as nice rewards for my study labour. I hope I will be able to pay my debt to them by my study results and their consequences.

Very special thanks belongs to my very special friends for providing me with their help, company and valuable teasing conversations; and special thanks to all the people I met in academic ambience and who learned me important, sometimes very inestimable, lessons.

As last but not least: thank you, my nerves and my patience, for standing with me to the very end!

Contents

1 Theoretical part

1.1 Bonding joints introduction

Adhesive bonding is the process of uniting materials with the aid of an adhesive, a substance capable of holding such materials together by surface attachment. There are two principal types of adhesive bonding, structural and non-structural.[1]

Structural adhesive is a term generally used to define an adhesive whose strength is critical to success of the assembly. This therm is usually reserved to describe adhesives with high shear strength and good environmental resistance. Structural adhesives are generally meant to be permanent, and they are not easily unbonded to provide product disassembly.[2]

Non-structural adhesives are adhesives with much lower strength and permanence. They are generally used used for temporary fastening or to bond weak substrates. Examples of non-structural adhesives are pressure-sensitive films, wood glue, hot melts, and elastomeric adhesives.[2]

Strength can be readily matched to the substrate and stress characteristics to which the bond will be subjected. Most adhesives and tapes perform better when the primary stress is tensile or shear. In most industrial applications, however, a combination of stresses are involved that may include cleavage and peel. In general, epoxies hold up best to harsh environments.

There are four basic types of load applicable to adhesive bonds, see fig. 1.1 for illustration.

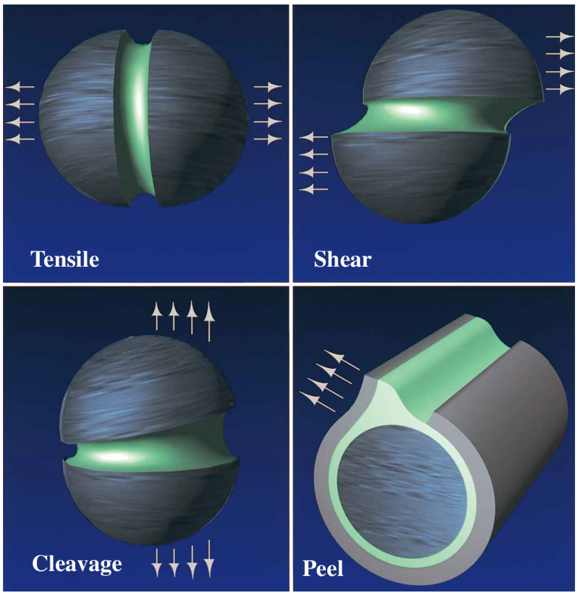

Figure 1.1: Basic types of bonding joints loading [3]

Tensile is pull exerted equally over the entire joint. Pull direction is straight and away from the adhesive bond.

Shear is pull directed across the adhesive, forcing the substrates to slide over each other.

Cleavage is pull concentrated at one edge of the joint, exerting a prying force on the bond. The other edge of the joint is theoretically under zero stress.

Peel is concentrated along a thin line at the edge of the bond where one substrate is flexible. The line is the exact point where an adhesive would separate if the flexible surface were peeled away from its mating surface. Once peeling has begun, the stress line stays out in front of the advancing bond separation.[3]

For enhancement of bond strength we need to know which attributes of the bond do influence the bond strength. Here are the four attributes on which the bond strength depends[4]:

- Adhesion
- Cohesion - strength of adhesive structure
- Wetting of surface
- Strength of bonded material (substrate)

Adhesives are means for creating part assemblies with a wide scale of use in industry and therefore a proper knowledge of adhesive properties is necessary. For it is propitious that when a property is tested, results from different test performers are comparable, standards for various testing methods were established. In the following section methods for measuring of adhesive bond properties related to three of the aforementioned attributes are described.

1.2 Normative testing of adhesive joints

The following text describes some of the main testing methods used for testing of adhesive bonds ("Normative references" is a section with list of standards related to particularly described standard). Some of the methods test interactions between adhesive and adherent material (strength of the bond assembly), some do test adhesive properties. For testing the adhesive properties cohesive failure of the bond is required and therefore a proper surface treatment shall be applied to secure this requirement. For enhancement of bond strength, adherent surfaces are prepared in many different ways. Preparation may be performed in accordance with producer's instructions or with EN 13887 standard, which describes many different possibilities of preparation for different materials.

By some of the testing methods, design information may be obtained, while the others do only provide informative results useful for comparison of adhesives or bond assemblies. The EN 15337 standard declares, that "Design data may be obtained by testing of particular combination of adhesive and adherent material which is to be used in the actual structure."[5]

Specimens for many of the testing methods are of a shape of stripes or shape similar to this. Many standards mention two preparation possibilities of testing specimens or semi-finishes, which are to bond specimens separately, or to cut them from bounded plates (milling and band-sawing are commonly used methods). Both of these possibilities should be equal and should give equal results (method of preparation should not influence the bond or progress of testing), however some methods require specimens prepared separately (if so, it is noted in description of respective standards).

When comparing results from different experiments, it is important to consider environmental conditions in which the experiments were performed (temperature, humidity, etc.) for it is adhesives nature that its behaviour has a close relation with its service environment.

Generally applies that values obtained by following methods shall be used as comparative information in order to compare different adhesives used for same adherent materials, or different adherent materials used with one type of adhesive.

1.2.1 Peeling tests (peel strength, peel resistance, wet peel resistance)

The following normatives describe methods used for measuring of peel resistance (also referred to as peel strength) and its modification - wet peel resistance. In EN 1464:2010 the peel resistance is defined as "an average force per unit specimen width, measured along the bond line, required to separate progressively the two members of a bonded test specimen under specified conditions of test"[6]. The peel resistance may be measured under different peeling angles.

1.2.1.1 EN 1464 – Adhesives – Determination of peel resistance of adhesive bonds – Floating roller method [6]

Normative references: EN 923:2005, EN ISO 291, EN ISO 10365

This normative states a method used for measuring of a peel resistance or a wet-peel resistance of adhesive bond if one adherent is rigid while the other one is flexible. It can be performed under any conditions required to test. This method is said to give the most constant numerical data from all of the peeling methods. For the testing procedure, specimen is arranged in a device used for application of loading force using a "peel test fixture" as shown in fig. 1.2. As different materials have different behaviour when bent, it cannot be expected, that every type of flexible adherent will align perfectly to a roller shape. During the testing a force against a crosshead movement (distance peeled) are to be recorded and charted. From the curve in chart an average, minimal and maximal peeling force are stated, according to instructions in the standard. The average force is used for determination of peel resistance in newtons per millimetre specimen width [N/mm].

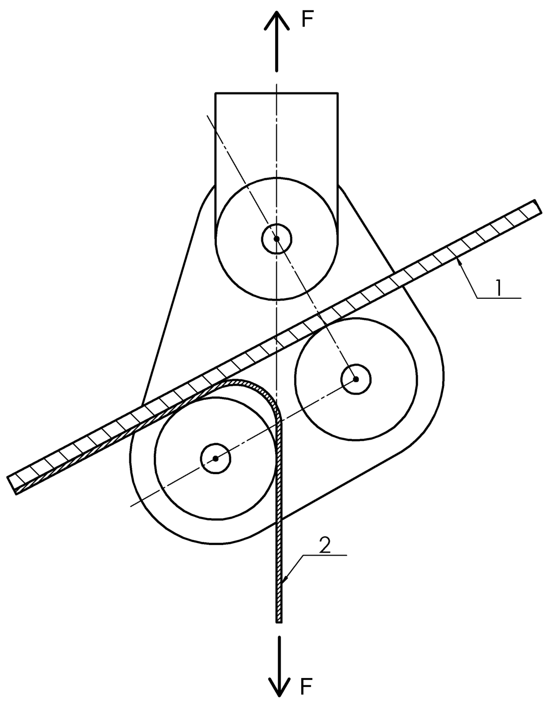

Figure 1.2: Floating roller method - peel test fixture and specimen adjustation; 1-rigid adherent, 2-flexible adherent (according to EN 1464)

Wet-peel resistance is measured in the same way as the "dry" peel resistance. The only difference is that after a specified peeled length a wetting agent dissolved in water is applied to the crack opening. (Wetting agent is a substance which lowers a surface tension of liquid. Liquid can than spread on a surface more easily and with a lower contact angle, due to a better wetting. [7])

1.2.1.2 EN ISO 11339 – Adhesives – T-peel test for flexible-to-flexible bonded assemblies [8]

Normative references: ISO 291, ISO 10365, ISO 17212

This normative states a method used for measuring of a peel resistance of adhesive bond. It is used when both adherents are flexible, nevertheless one adherent can be more flexible than the other one. It was originally developed for metal adherents, but can be used for adherent of other materials (if applicable) as the standard provides information how to adjust specimens for various materials. Results obtained by this method shall not be used for design calculations, only as reference information. Specimens for this method (see fig. 1.3) are prepared from bonded adherent plates. Once the bond is stable, the unbounded ends of specimen are bent in opposite ways until they are perpendicular to a bond line, forming a T shape assembly. Radius of bending is not specified. If one adherent is less flexible than the other, the less flexible one shall be adjusted in a moveable grip clamp.

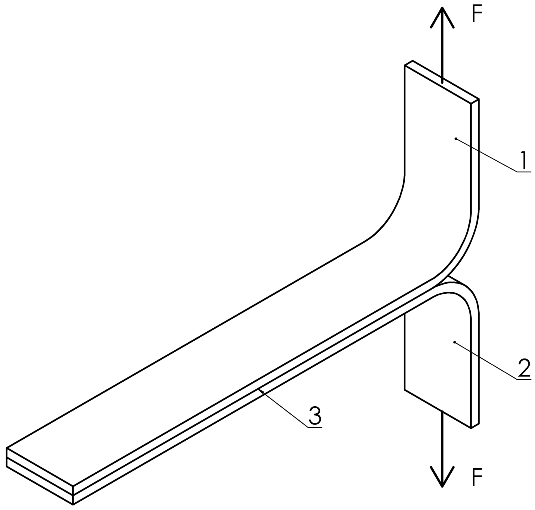

Figure 1.3: T-Shape specimen; 1-more flexible adherent, 2-more rigid adherent, 3-bond line (according to ISO 11339)

Applied force versus distance of grip (grip movement) is recorded. Angle between the bond line and force applied is not fixed. Loading force versus a grip movement are recorded and charted. From the curve obtained in the chart an average, minimal and maximal peeling force are determined according to instructions in the standard. The average force is used for determination of peel resistance in newtons per 100 millimetre specimen width [N/100mm].

1.2.2 Adhesion

1.2.2.1 EN 1966 – Structural adhesives – Characterization of a surface by measuring adhesion by means of the three point bending method [9]

Normative references: EN 923:2005, EN 10025-2, EN 13887, EN ISO 291, EN ISO 9142, EN ISO 10365, ISO286-1

This normative may be used for determining of an ability of adhesive to adhere to a surface. By this method can be decided, to which adherent or surface finish the adhesive has the best adhesion, or which adhesive has the best adhesion to one specific surface. This testing method is not suitable for film adhesives. Only reference values for specimen comparison can be obtained by this method, not magnitude of adhesive (eventually cohesive) forces. According to a failure pattern can be determined, whether the twosome of surfaces and an adhesive will provide an adhesive or cohesive failure of bond. For this method an adhesive is cured in special form described in the normative. It is given the shape of block bonded to a tested surface. Between the adhesive and surface can only be one interfacial zone. In specific cases the block can be replaced by a bonded brace of the same material as the adherent. When tested, the load is applied perpendicularly to the adherent surface in the middle between both supports (see fig. 1.4), force against deflection is recorded and charted (see fig. 1.5).

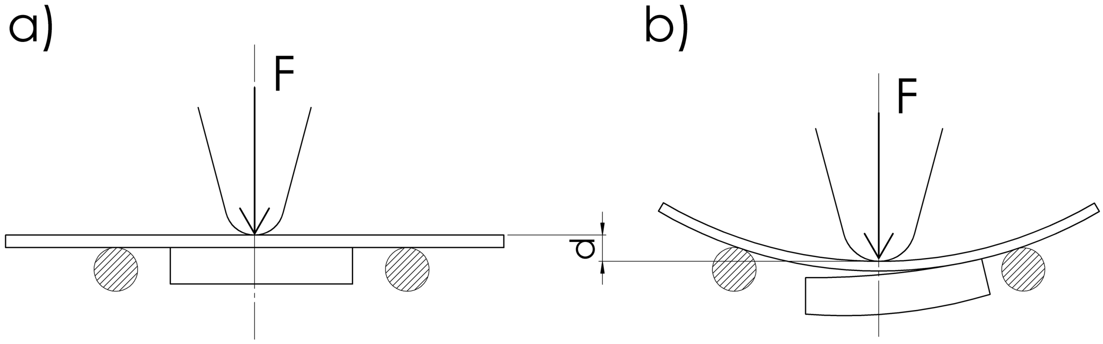

Figure 1.4: a) Testing assembly b) Specimen under load with adhesive bond failure; F-applied force, d-deflection of specimen (according to EN 1966)

For the result evaluation bare adherent (with surface finish if this is tested) without the adhesive block must be tested and force with deflection recorded. When charted in one chart (see fig. 1.5), the result of this testing is the difference of values of force at initial bond destruction and force applied to the bare adherent at the same deflection. The maximal steepness represents a sameness of tested specimens

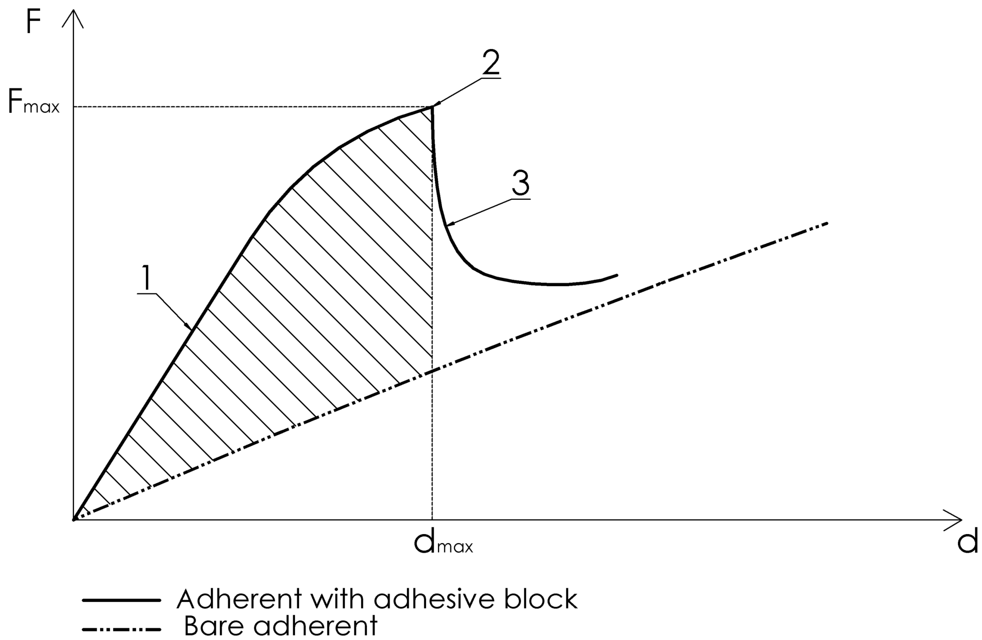

Figure 1.5: Chart for results evaluation; 1-maximal steepness, 2-maximal force and deflection, 3-fracture propagation (according to EN 1966)

1.2.3 Shear (shear strength, shear strain, shear modulus)

The following standards describe various methods for determination of adhesive shear strength τ, using different samples and loading principles. Shear strain γ and shear modulus G of adhesive are also determined. The standards provide all necessary calculation formulas.

Shear stress – force applied parallel to flat adhesive joint, divided by the bond area of the joint.
Shear strength – maximum shear stress sustained by an adhesive joint during a shear test

1.2.3.1 EN 1465 – Adhesives – Determination of tensile lap-shear strength of bonded assemblies [10]

Normative references: EN 13887, EN ISO 291, EN ISO 527-1, EN ISO 10365

This normative describes a method for determination of shear strength of adhesive, using two overlapped rigid adherents (see fig. 1.6) loaded by tensile stress, which leads to shear loading of adhesive. Results obtained by this method shall not be used for design calculations, only as reference values used for specimen comparison.

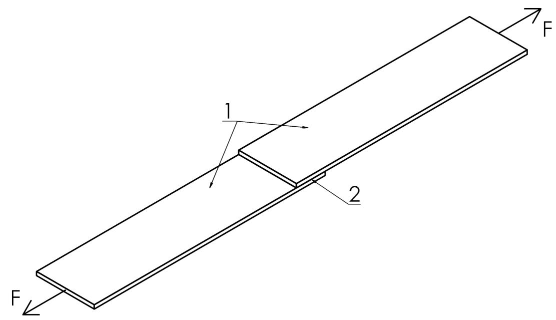

Figure 1.6: Specimen design; 1-adherents, 2-bond line (according to EN 1465)

Specimens used for this testing may be prepared separately (stripes bounded together) or cut from bonded plates. Because the ends of stripes which are clamped and pulled are not collinear, the stripes would get deflected and loading force would not be collinear with a bond line (or bond plane). Therefore the adhesive would not be loaded by pure shear. To prevent this compensation plates are adjusted to clamps, ensuring the stripes do not deflect under the load. When loading the specimen the highest recorded force sustained by the bond is considered as the force needed for the bond breakage. This force expressed in newtons is divided by bond area expressed in square millimetres, obtaining the shear strength expressed in megapascals [MPa] [N/mm2].

1.2.3.2 EN ISO 13445 –Adhesives – Determination of shear strength of adhesive bonds between rigid substrates by the block-shear method [11]

Normative references: ISO 291:1997, ISO 7500-1, ISO 10365:1992, EN 13887

This normative describes a method for determination of a shear strength τ of adhesive bond using two rigid blocks with an overlap bond. This method is suitable for metal, plastic, ceramic, glass, magnet moldings and wood materials as well as for their combinations.
Specimens for this method consist of two rigid adherent blocks. These must be rigid enough so that no adherent deformation occurs during the test. Specimen is adjusted in a testing assembly (see fig. 1.7) so that one block is placed in specimen-holding block and secured with a toggle clamp, which ensures that the specimen does not move inappropriately during the test. Shearing tool is put on the other specimen block. Load is applied to the specimen via the holding block and shearing tool.

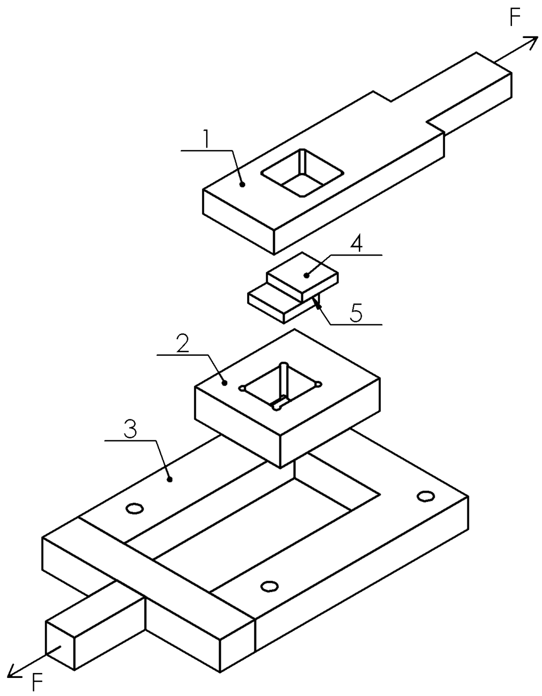

Figure 1.7: Testing assembly; 1-shearing tool, 2-adapter for small specimens, 3-holding block, 4-specimen, 5-bond line (according to EN ISO 13445)

Maximal force needed for a bond failure is recorded and used for calculation of the bond shear strength, expressed in megapascals [MPa]. Failure pattern (adhesive/ cohesive failure) shall also be determined.

1.2.3.3 EN 14869-1 – Structural adhesives – Determination of shear behaviour of structural bonds – Part 1: Torsion test method using butt-bonded hollow cylinders [12]

Normative references: EN 923:2005+A1:2008, EN 13887, EN ISO 291, EN ISO 10365

This normative describes a method for determination of shear strength τ of adhesive, using two coaxial hollow cylinders bound in an annular butt joint. Also a shear strain γ, angular displacement α and shear modulus G of adhesive are determined, all necessary calculation formulas are provided in the normative. For validity of this method a cohesive bond failure is required and therefore an appropriate surface treatment shall be applied, to ensure such failure. For specimen (see fig. 1.8) preparation two hollow cylinders are bound together by their bases. Special jigs, spacers and Polytetrafluorethylen (PTFE) plug are used to ensure coaxial connection of both cylinders and that no adhesive will run out of the joint during the curing time as well as proper adhesive layer thickness.

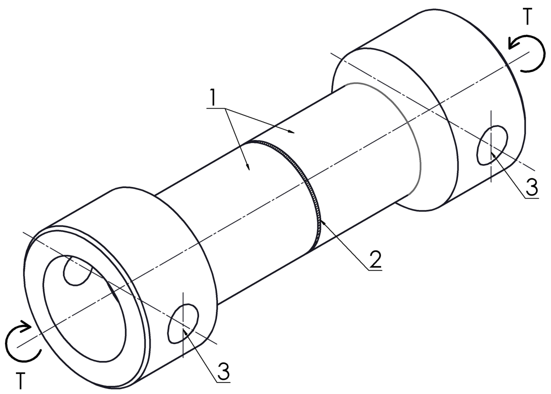

Figure 1.8: Specimen design; 1-adherent (hollow cylinders), 2- bond line, 3-holes for fastening bolts (according to EN 14869-1)

The cylinders are loaded by torque and the torque-displacement data are recorded and charted. These data are than used for calculations of desired variables. Shear strength τ expressed in megapascals [MPa] is calculated from highest loading force recorded (highest force sustained by adhesive).

1.2.3.4 EN 14869-2 – Structural adhesives – Determination of shear behaviour of structural bonds – Part 2: Thick adherents shear test [13]

Normative references: EN 923:2005+A1:2008, EN 13887, EN ISO 291, EN ISO 10365, ISO 683-11, ISO 1052, ISO 4995

This normative describes a method for determination of shear strength of adhesive, using two thick, rigid steel adherents bound in a single lap joint loaded by a tensile force, determining in adhesive loaded by shear stress. For validity of this method a cohesive bond failure is required and therefore an appropriate surface

treatment shall be applied, to ensure such failure.
Short overlap length of specimen should ensure the most uniform distribution of shear stress possible and minimize other stress states which initiate failure. Using data obtained by this method shear strength τ, shear strain γ, shear modulus G of adhesive are stated, all necessary calculation formulas are provided by the normative. Specimens used for this method are of two equal designs (see fig. 1.9).

a) Flat-ended adherents are prepared from two metal strips bound together. Holes for holding pins are drilled and the overlap zone is delineated by milling two grooves. The specimens may be prepared from pre-bound panels or separately. It may be good to use a milling tool with radius or chamfer at its nose, which will form a radius or fillet end of bond, reducing the strain concentration as in b).

b) Stepped adherents are machined prior to bounding. PTFE or steel (covered in release agent) stripes shall be inserted in-between adherents in order to secure proper length and shape of bond. Stripes are recommended to have 45° tapered edge, which forms a triangular fillet at a bond edge, reducing the strain concentration at the bond edge.

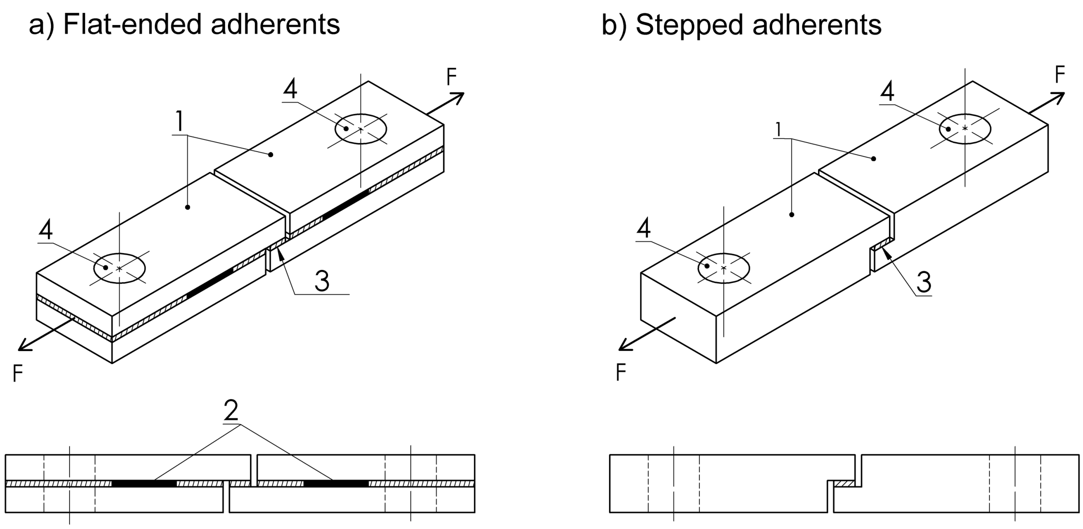

Figure 1.9: Specimen design; 1-adherents, 2-shim (optional), 3-bond line, 4-holes for fastening bolts (according to EN 14869-2)

Specimen is adjusted in testing machine using holding pins, which allows the specimen to turn itself well aligned with both clamps, ensuring that the specimen is not loaded with any additional loads. When the tensile load is applied to the specimen, the bond is loaded by shear, force against displacement is recorded. Method uses extensometers for measuring of shear displacement. These data are than used for determination of tensile strength τ expressed in megapascals [MPa] calculated from highest loading force recorded, and other desired variables using given computation formulas. Shear stress – shear strain curve may also be obtained. The specimen in a) has a lower bending stiffness than the solid one in b), therefore the a) will have a higher peel stresses at the adhesive ends. However this difference causes only trivial deviation in comparative results.

1.2.3.5 EN 15337 – Adhesives –Determination of shear strength of anaerobic adhesives using pin-and-collar specimens [5]

Normative references: EN ISO 7500-1

This normative describes a method for determination of shear strength τ of anaerobic-curing liquid adhesives used for retaining of cylindrical assemblies pin-and-collar type, or locking and sealing threaded fasteners, however may also be used for other adhesives. Results obtained by this method are of reference nature, useful for ranking and quality control of adhesives. It cannot be expected to fully reflect the bond performance in service and to provide numerical data for design purposes in general. Generally low-grade carbon steel is used for the adherents, unless other materials are required to test. Design data may be obtained by testing of particular combination of adhesive and adherent material which is to be used in the actual structure.
Specimens for this testing (see fig. 1.10) are prepared by bonding a cylindrical pin and a slip collar, where adhesive is applied to the common lateral surface.

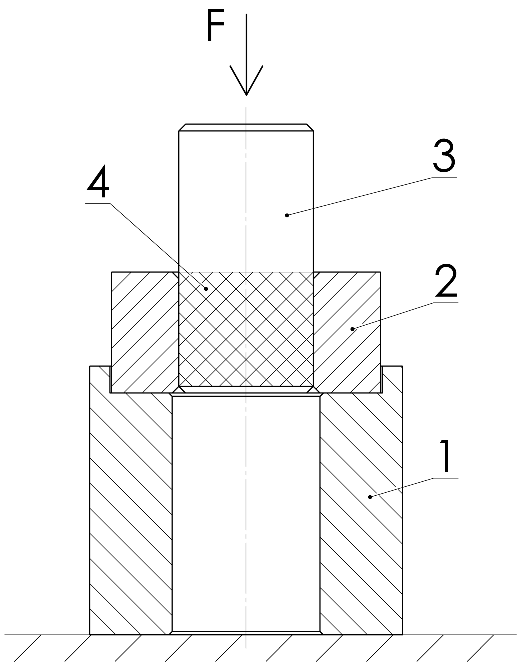

Figure 1.10: Testing assembly; 1-specimen support, 2-collar, 3-pin, 4-adhesive coated area (according to EN 15337)

For testing the specimen is placed on a specimen support and loaded by compressive force perpendicular to the specimen base. The maximal loading force sustained by the bonded assembly is recorded and used for calculation of static shear strength, determined as the maximal force in newtons divided by lateral surface area of the pin in square millimetres, expressed in megapascals [MPa].

1.2.4 Impact (impact resistance, impact strength, impact value)

The following standards specify methods for determination of impact resistance (also announced to as impact strength or impact value), which can be expressed as force, needed to cause a bond failure, per specimen width or energy absorbed by specimen during the impact, depending on method used for determination.

1.2.4.1 ISO 9653 – Adhesives – Test method for shear impact strength of adhesive bonds [14]

Normative references: ISO 291, ISO 4588, ISO 9142, ISO 10365, ISO 13895

This standard specifies method for determination of shear impact strength of adhesive bond expressed in joules (energy) per square meter adhesive bond area. The specimen (see fig. 1.11) used for testing consists of two blocks of specified dimensions bond together. Materials of specimens may be metal, wood, plastic or combination of two of these materials. When bond is settled, the specimen is placed in a vice and fastened using a clamp screw. The whole assembly shall be adjusted so that in a moment of strike the pendulum strikes at its highest velocity and a striking face of pendulum and impact face of specimen are parallel. Energy absorbed during the bond destruction is read and divided by bonded area, obtaining the impact value expressed in joules per square meter bond area [J/m2]. The normative requires the values reported to the nearest 100 J/m2.

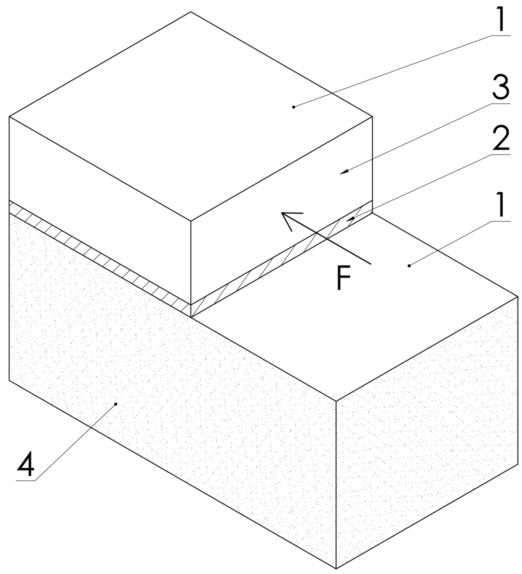

Figure 1.11: Specimen design; 1-adherents, 2-bond line, 3-impact face, 4-faces fastened in vice (according to ISO 9653)

In case of metal adherents the specimen blocks may be reused if no deformation occurs, for other adherent materials such possibility is not specified.

1.2.4.2 ISO 11343 – Adhesives – Determination of dynamic resistance to cleavage of high-strength adhesive bonds under impact conditions – Wedge impact method [15]

Normative references: ISO 291:1997, ISO 10365:1992, EN 13887

This standard describes a method for measuring of a dynamic resistance to cleavage of adhesive bond using a wedge impact on an adhesive bond line along its length, causing the bond to fail in a peeling mode. This methods use is suitable for high-strength adhesives, used for bonding of two metallic adherents. Nevertheless it does not provide design information.
Specimens (see fig. 1.12) for this method are prepared separately. Two stripes are bond together at one end, than bent so an adjustation of a wedge situated at a wedge support frame, does not interfere with the bond and provide a correct trajectory of the wedge. Using a spacer, clamping plate and retaining bolt the specimen is fastened. Striking faces of the wedge support frame are struck by pendulum (falling-weight or servo-hydraulic-impact machines are also possible to use), forcing the wedge through the adhesive bond, causing the plates peel separate. Force-time or force-displacement data are recorded and used for determination of average force, which is than divided by specimen width to obtain the cleavage resistance expressed in kilonewtons per meter specimen width [kN/m].

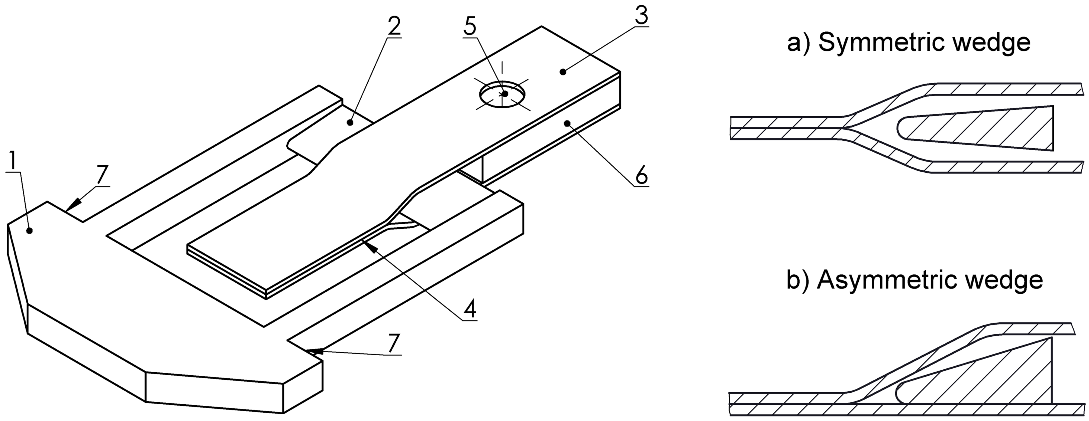

Figure 1.12: Specimen adjustation, symmetric and asymmetric wedge; 1-wedge support frame, 2-wedge, 3-specimen, 4-bond line, 5-retaining-bolt hole, 6-spacer, 7-striking face (according to ISO 11343)

When both adherents have same properties a symmetric wedge is used. If the adherents provide different thickness or different modulus, an asymmetric wedge is used, and the thicker or tougher adherent is aligned with its bottom (flat) plane.

1.2.5 Torsion (torque strength)

1.2.5.1 EN 15865 – Adhesives – Determination of torque strength of anaerobic adhesives on threaded fasteners [16]

Normative references: EN 20898-2, EN ISO 291, EN ISO 898-1

This standard specifies method for testing of torque strength of threaded assemblies supported by an adhesive bond in thread connection. The testing gives values used for comparison of securing effects of adhesives used for thread assemblies, when primarily meant for testing of anaerobic adhesives. Assemblies used for the testing (see fig. 1.13) consist of a normalized bolt, normalized nut, adhesive and spacer sleeve (for seated assemblies). During the testing torques at different stages of unscrewing a nut form a bolt are measured. The most important may be a torque needed for an initial breakage of the bond. Its value may be useful for design in industry. Torque strengths of unseated (without spacer sleeve) and seated (preloaded with an input torque) assemblies are measured as well as torque strengths of assemblies without the adhesive bond support, so an assembly strength enhancement is to be clearly visible.

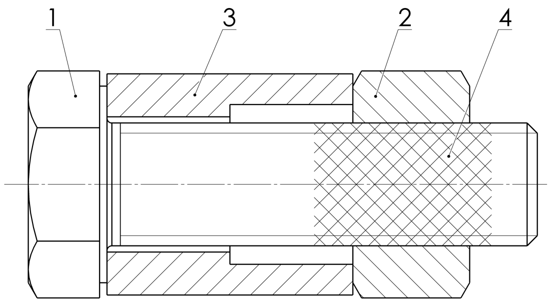

Figure 1.13: Testing assembly; 1-bolt, 2-nt, 3-spacer sleeve, 4-adhesive coated area (according to EN 15865)

1.2.6 Tensile (tensile strength)

1.2.6.1 Tensile EN 15870 – Adhesives – Determination of tensile strength of butt joints [17]

Normative references: EN 13887, EN ISO 291, EN ISO 7500-1, EN ISO 10365

This normative describes a method for determination of tensile strength σ of adhesive bond, when under tensile stress, expressed as force needed for bond failure. This method may be applied for any type of adhesive under conditions set by this standard, or any other desired environmental or other conditions.
Adherents of a rod shape are bound using the tested adhesive by their bases. Jigs may be used to ensure a collinearity of the rods. Tensile load is than applied parallel to a longitudinal axis of rods and force sustained by the bond is determined. Adherents used for this testing must be of a tough nature (so that no deformation

could be observed during the testing), must be of defined diameters and must have higher tensile strength than tested adhesive.
The normative states only evaluation of tensile strength as an average force needed for bond breakage, given that for inter-laboratory data comparison specimens must be of prescribed dimensions. If this force was divided by the bond area, a tensile strength σ expressed in Pa [N/m2] or MPa [N/mm2] may be obtained. Therefore these values may be used for inter-laboratory data comparison without any need for abidation of prescribed specimen dimensions, providing easier sample preparation. However this idea is not yet supported by the EN 15870 normative.

2 Experimental part

2.1 Objective

In this chapter I will compare six different adhesives provided by 3M Česko, spol. s r.o. company suitable for bonding of metal to thermoplastic, focus on shear strength of bond they provide when bonding aluminium stripe to polyethylene stripe, according to EN 1465.

Each adhesive will be used for samples with plasma treated polyethylene and non-treated polyethylene. Effect of plasma treatment will also be evaluated. With one exception the manufacturer recommends the use of adhesives for industry with plasma treated surfaces, even though they are suitable for use with thermoplastics.

2.2 Materials and adhesives used

Thickness of plates according to the EN 1465 standard should be 1.6 mm, but sheets of such thickness were difficult do find. Therefore sheets of different thickness were used. For the results obtained in this experiment are not meant for interlaboratory comparison, but only to compare the samples with each other, it is not necessary to comply with standardised thickness of samples.

2.2.1 Aluminium stripes

Aluminium sheet used for samples preparation was 1.5 mm thick aluminium sheet purchased from ALUPLUS a.s. company. Stripes for samples were cut according to diameters given by the EN 1465 standard, i.e. 25 x 100 mm. All stripes had their bonding area coarsened using a 80 grit sand paper.

Information provided by seller:
Alloy: EN AW-1050A (Al99,5)
State of material: H24 semi-hard
Chemical composition: EN 573-3
Mechanical properties: EN 485-1
Geometric tolerance: EN 485-4

2.2.2 Polyethylene stripes

Sheet used for samples preparation was 2mm thick, extruded, natur, high density polyethylene PE 300 from TITAN – MULTIPLAST s.r.o. company, secured, cut and plasma treated by SurfaceTreat, a.s. company. Stripes for samples were cut according to diameters given by the EN 1465 standard, i.e. 25 x 100 mm. More information about material used can be found in data sheet attached.

2.2.3 DP 8805 NS Green

Acrylic based, non-sagging, two-component adhesive with work-time of 3-5 minutes and time to structural strength of 8-10 minutes. After curing it has a chemical odour noticeable at close examination and tough, slightly elastic nature. It contains glass beads of 0,25 mm in diameter for bond line thickness control. According to a data sheet (see attachment) it has an excellent shear strength and high peel and impact strength. The flexible nature of adhesive has potential for use where slight vibration absorption is useful. If used for bonding of plastic materials, bond area surface shall be treated (for example chemical treatment or plasma). [18]

2.2.4 DP 8005 Off-White

Acrylic based, two-component adhesive with work-time of 2.5-3 minutes and time to structural strength of 8-24 hours. After curing it has a very low odour noticeable at close examination, tough and slightly elastic nature. Adhesive contains glass beads of 0,2 mm in diameter for bond line thickness control. The flexible nature of adhesive has potential for use where slight vibration absorption is useful. This adhesive is suitable for bonding of low surface energy plastics without any special surface treatment. [19]

2.2.5 DP 620 NS Black

Urethane based, non-sagging, two-component adhesive with work-time of 20 minutes and time to structural strength of 48 hours. After curing it has a very low odour noticeable at close examination and hard, brittle nature. If used for bonding of plastic materials, bond area surface shall be treated (for example chemical treatment or plasma). According to a data sheet it has a high tensile strength and resists temperatures in wide range. [20]

2.2.6 DP 760 White

Epoxy based, two-component adhesive with work-time of 60-80 minutes. Time to structural strength is not specified in data sheet, but according to information mentioned, it shall be a few hours. After curing it has no odour noticeable at close examination and hard, brittle nature. If used for bonding of plastic materials, bond area surface shall be treated (for example chemical treatment or plasma). It is designed for high temperature resistance. [21]

2.2.7 DP 190 Gray

Epoxy based, two-component adhesive with work-time of 90 minutes and time to structural strength of 7 days. After curing it has a very low odour noticeable at close examination, tough and slightly elastic nature. If used for bonding of plastic materials, bond area surface shall be treated (for example chemical treatment or plasma). According to a data sheet it has a high shear and peel strength. The flexible nature of adhesive has potential for use where slight vibration absorption is useful.[22]

2.2.8 DP 100 Plus Clean

Epoxy based, two-component adhesive with work-time of 3 minutes and time to structural strength of 48 hours. When applied it has a strong odour. After curing it has a very low odour noticeable at close examination and the adhesive has a tough, elastic nature, which is emphasized also in data sheet. With flexibility the adhesive is said to have a high peel and shear strength and its flexible nature has potential for use where slight vibration absorption is useful. If used for bonding of plastic materials, bond area surface shall be treated (for example chemical treatment or plasma). [23]

2.3 Samples preparation

2.3.1 Measurement of polyethylene surface energy:

the treatments aim is to increase a surface energy of polyethylene, making it more suitable for adhesive bonding. Surface energy was measured on plasma treated and non-treated surface to evaluate the enhancement of bonding conditions. This measurement was performed using Arcotest test inks (see fig. 2.1).

Figure 2.1: Arcotest test inks for measuring of surface energy

Fig. 2.2 shows the enhancement of surface energy after plasma treatment. Before the treatment (PE) the surface had energy of 28 mN/m (the ink forms nice even line). After the treatment (PPE) the surface energy raised to 48 mN/m, which should lead to a better adhesion between the adhesive and polyethylene.

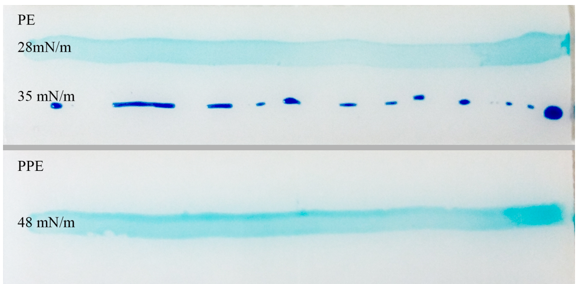

Figure 2.2: Measurement of surface energy of non-treated polyethylene (PE) and plasma treated polyethylene (PPE)

2.3.2 Plasma treatment conditions:

displacement speed of plasma jet: 50 mm/s
air flow: 20 l/min
distance between jet and treated surface: 8 mm

2.3.3 Bonding and adhesive curing:

Before bonding or plasma treatment all bond areas of aluminium and polyethylene were cleaned with isopropyl alcohol. Adhesives were applied using applicator provided by 3M company and mixing nozzles, so adhesive components were properly mixed in correct mixing ratio. During the bonding process, adhesive was applied to both surfaces and than connected together. To secure the bond line to be even and keep the specimen in the right shape the polyethylene was underlayed with another aluminium stripe, so the PE stripe was horizontal during the cuing process (see fig. 2.3)

Figure 2.3: Samples adjustation for curing (on the left side the aluminium underlay can be seen)

All adhesives were cured under same conditions, i.e. at a room temperature for 18 days to ensure that even the adhesive with longest time to structural strength is cured properly.

2.3.4 Tensting conditions

All samples were tested at room temperature, i.e. 21°C.

Three samples were tested with crosshead speed of 10 mm/min, others with speed of 50 m/min. That was because the normative does not set the crosshead speed, that shall be used, but time within which the sample shall be destructed, so I needed to find suitable speed to achieve this testing condition.

I also performed testing of PE under different crosshead speeds to find out, how much does the speed influence the strength of the material. If the influence was significant, it would have been better to test samples at higher crosshead speed for the PE substrate would have sustained higher loading force and the adhesive limits may have been tested more thoroughly, because the substrate failure would not have appeared at lower loading force values. Nevertheless it showed up (see chart at fig. 3.13 in attachment), that the rise is not very significant, so testing of all adhesives was than performed at crosshead speed of 50 mm/min.

2.4 Measured values processing

2.4.1 DP 8805 NS Green

Table 2.1: Data from shear test of DP 8805 with non-treated polyethylene; based on chart at fig. 3.1 in attachment.

Non-treated polyethylene DP 8805					
Sample nr.	1	2*[1]	3	4	5
Crosshead speed [mm/min]	50	50	50	-[2]	-
Maximal force [N]	320	260	310	-	-
Shear strength [N/mm^2]	1.02	0.83	0.99	-	-
Failure pattern[3]	AF	AF	AF	-	-

Avg. max. force: 296.67 N

Avg. shear strength: 0.95 N/mm^2

Table 2.2: Data from shear test of DP 8805 with plasma treated polyethylene; based on chart at fig. 3.2 in attachment.

Plasma treated polyethylene DP 8805					
Sample nr.	1	2*	3	4	5
Crosshead speed [mm/min]	50	50	50	50	50
Maximal force [N]	630	800	710	890	825
Shear strength [N/mm^2]	2.02	2.56	2.28	2.85	2.64
Failure pattern	AF	AF	AF	AF	AF

Avg. max. force: 771 N

Avg. shear strength: 2.47 N/mm^2

After plasma treatment the average force sustained by samples grew up to 159,9%.

[1]"*" marked samples were tested with use of compensation plates at both sides of sample. Samples tested with the plates proved to be lesser strong, or the plate had no influence at sample strength (can bee seen at graphs from tests - samples with "po" suffix), therefore the general amount of samples was tested without the plates

[2]"-" marked samples were damaged when an excess adhesive was being removed after curing. It happened in way that adhesive got clearly peeled from polyethylene. Nevertheless, it only happened with untreated polyethylene, which shall not be used with all but one of these adhesives for industry purposes. None of the samples with plasma treated polyethylene were damaged during manipulation.

[3]AF - adhesion failure pattern, CF - cohesion failure pattern, SF - substrate failure pattern (in this experiment it was always polyethylene failure)

2.4.2 DP 8005 Off-White

Table 2.3: Data from shear test of DP 8005 with non-treated polyethylene; based on chart at fig. 3.3 in attachment.

Non-treated polyethylene DP 8005					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	50
Maximal force [N]	1100	1110	1100	1080	1110
Shear strength [N/mm^2]	3.53	3.56	3.53	3.46	3.56
Failure pattern	SF	SF	SF	SF	SF

Avg. max. force: 1100 N

Avg. shear strength: 3.53 N/mm^2

Table 2.4: Data from shear test of DP 8005 with plasma treated polyethylene; based on chart at fig. 3.4 in attachment.

Plasma treated polyethylene DP 8005					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	50
Maximal force [N]	1140	1110	1110	1120	1140
Shear strength [N/mm^2]	3.65	3.56	3.56	3.59	3.65
Failure pattern	SF	SF	SF	SF	SF

Avg. max. force: 1124 N

Avg. shear strength: 3.60 N/mm^2

In case of this adhesive there is no point in evaluating growth of bond strength because all samples failed in polyethylene. Producer says the adhesive is suitable for bonding of low surface energy plastics without any prior surface preparation, which is proved by fact, that all non-treated samples failed in bonded substrate, i.e. bond was stronger than bonded material.

2.4.3 DP 760 White

Table 2.5: Data from shear test of DP 760 with non-treated polyethylene; based on chart at fig. 3.5 in attachment.

Non-treated polyethylene DP 760					
Sample nr.	1	2	3*	4	5
Crosshead speed [mm/min]	50	10	50	50	50
Maximal force [N]	210	210	48 - X	118	182
Shear strength [N/mm^2]	0.67	0.67	0.15	0.38	0.58
Failure pattern	AF	AF	AF	AF	AF

Avg. max. force: 180 N

Avg. shear strength: 0.58 N/mm^2

Table 2.6: Data from shear test of DP 760 with plasma treated polyethylene; based on chart at fig. 3.6 in attachment.

Plasma treated polyethylene DP 760					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	10	50	50	50	50
Maximal force [N]	958	1140	980	1080	800
Shear strength [N/mm^2]	3.07	3.65	3.14	3.46	3.56
Failure pattern	SF	SF	AF+CF	SF	AF

Avg. max. force: 992 N

Avg. shear strength: 3.38 N/mm^2

After plasma treatment the average force sustained by samples grew up to 451%. The rise may be higher, but could not have been measured because of the strength of polyethylene. It may be measured with use of polyethylene with bigger cross-section. Nevertheless I am of meaning that the measured value is close to this adhesives limit, since both adhesion and substrate failure appeared, within tested samples lot.There is a difference of 200 N between highest and lowest strength values where substrate failure occur. It may be caused by notches on PE stripes, which decreased total tensile strength of particular stripes.

2.4.4 DP 620 NS Black

Table 2.7: Data from shear test of DP 620 NS with non-treated polyethylene; based on chart at fig. 3.7 in attachment.

Non-treated polyethylene DP 620 NS					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	-
Maximal force [N]	150	23 - X[4]	81 - X	171	-
Shear strength [N/mm^2]	0.48	0.07	0.26	0.55	-
Failure pattern	AF	AF	AF	AF	-

Avg. max. force: 160.5 N

Avg. shear strength: 0.52 N/mm^2

Table 2.8: Data from shear test of DP 620 NS with plasma treated polyethylene; based on chart at fig. 3.8 in attachment.

Plasma treated polyethylene DP 620 NS					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	50
Maximal force [N]	1120	1110	860	980	1070
Shear strength [N/mm^2]	3.59	3.56	2.76	3.14	3.43
Failure pattern	SF	SF+AF[5]	SF	SF	SF

Avg. max. force: 1028 N

Avg. shear strength: 3.30 N/mm^2

After plasma treatment the average force sustained by samples grew up to 540.5%. The rise may be higher, but could not have been measured because of the strength of polyethylene. It may be measured with use of polyethylene with bigger cross-section.

[4] I discharge "X" marked samples from statistics, because the values diametrically differ from the other two samples which have higher and close strength values. The discharged samples may have been imperceptibly damaged during a manipulation and adhesive excess removing.

[5] Adhesive part of failure appeared so that polyethylene peeled from half of bond area, but the other half stayed intact, which led to substrate failure.

2.4.5 DP 190 Gray

Table 2.9: Data from shear test of DP 190 with non-treated polyethylene; based on chart at fig. 3.9 in attachment.

Non-treated polyethylene DP 190					
Sample nr.	1	2*	3	4	5
Crosshead speed [mm/min]	50	50	50	50	-
Maximal force [N]	495	240 - X	448	420	-
Shear strength [N/mm^2]	1.59	0.77	1.44	1.35	-
Failure pattern	AF	AF	AF	AF	-

Avg. max. force: 454 N

Avg. shear strength: 1.46 N/mm^2

Table 2.10: Data from shear test of DP 190 with plasma treated polyethylene; based on chart at fig. 3.10 in attachment.

Plasma treated polyethylene DP 190					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	10	50	50	50
Maximal force [N]	1130	1010	1060	1090	1100
Shear strength [N/mm^2]	3.62	3.24	3.40	3.49	3.53
Failure pattern	SF	SF	AF	SF	SF

Avg. max. force: 1078 N

Avg. shear strength: 3.46 N/mm^2

After plasma treatment the average force sustained by samples grew up to 137.4%. The rise may be higher, but could not have been measured because of the strength of polyethylene. It may be measured with use of polyethylene with bigger cross-section. Nevertheless I am of meaning that the measured value is close to this adhesives limit, since both adhesion and substrate failure appeared, within tested samples lot. It may be even closer to limit, than the DP 760 adhesive, for adhesion and substrate failures have magnitudes of load sustained much closer to each other.

2.4.6 DP 100 Plus Clean

Table 2.11: Data from shear test of DP 100 Plus with non-treated polyethylene; based on chart at fig. 3.11 in attachment.

Non-treated polyethylene DP 100 Plus					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	-
Maximal force [N]	600	530	380	285	-
Shear strength [N/mm^2]	1.92	1.70	1.22	0.91	-
Failure pattern	AF	AF	AF	AF	-

Avg. max. force: 448.8 N

Avg. shear strength: 1.44 N/mm^2

Table 2.12: Data from shear test of DP 100 Plus with plasma treated polyethylene; based on chart at fig. 3.12 in attachment.

Plasma treated polyethylene DP 100 Plus					
Sample nr.	1	2	3	4	5
Crosshead speed [mm/min]	50	50	50	50	50
Maximal force [N]	1160	870	930	1150	1080
Shear strength [N/mm^2]	3.72	2.79	2.98	3.69	3.46
Failure pattern	SF	AF	AF	SF	SF

Avg. max. force: 1038 N

Avg. shear strength: 3.33 N/mm^2

After plasma treatment the average force sustained by samples grew up to 131.3%. The rise may be higher, but could not have been measured because of the strength of polyethylene. It may be measured with use of polyethylene with bigger cross-section. Nevertheless I am of meaning that the measured value is close to this adhesives limit, since both adhesion and substrate failure appeared, within tested samples lot.

2.5 Measured values summary

Table 2.13: Measured values summary

Adhesive	DP 8805	DP 8005	DP760	DP620	DP 190	DP 100
Samples with on-treated polyethylene PE						
Max. sustained force [N]	320	1100	210	171	495	600
Avg. sustained force [N]	296.67	1100	180	160.5	454	448.8
Failure pattern - max. force	AF	SF	AF	AF	AF	AF
Samples with plasma treated polyethylene PPE						
Max. sustained force [N]	890	1140	1140	1120	1130	1160
Avg. sustained force [N]	771	1124	992	1028	1078	1038
Failure pattern - max. force	AF	SF	SF	SF	SF	SF
Avg. force growth [%]	159.9	-	451	540.5	137.4	131.3

Table 2.14: Indicative adhesive prices (according to producers website[6])

Adhesive	DP 8805	DP 8005	DP760	DP620	DP 190	DP 100
Cartige volume [ml]	45	38	50	50	50	50
Price[7] [Kč]	518	772	387	687	536	549
Price per ml [Kč/ml]	11.5	20.3	7.7	13.7	10.7	11

[6]http://www.g3.cz/katalog/lepidla-3m
[7]including VAT

3 Results and discusion

The theoretical part contents a summary of twelve testing methods used for testing of different properties of adhesive bonds. Each method has a description of testing principle, evaluation of tested property, illustration images are also present.

In practical part an experiment according to EN 1465 standard was performed. The objective was to compare shear performance of six adhesives suitable for adhesive bonding of (thermo)plastics to metals. Amongst them all three types of adhesive bases were present (epoxy, urethane, acrylic). As substrates polyethylene and aluminium were chosen. Each adhesive was tested with non-treated and plasma treated polyethylene surface in order to determine the influence of plasma treatment at bond strength.

As least suitable adhesives proved to be the DP 8805 with average sustained loading force with plasma treated polyethylene of 771 N, it also formed easily bond damaged when removing the excess adhesive.

The DP 760 with 992 N of average loading force sustained by plasma treated samples does not provide much strong bond. The DP 620 with 1028 N of average sustained loading force and 3 out of 5 samples providing the adhesion failure at plasma treated samples show the adhesive was close to its limits. Therefore I consider these two as not very suitable for bonding the twosome of chosen substrates.

The DP 190 with 1078 N and DP 100 with 1038 N of average sustained force by samples with plasma treated polyethylene proved to be relatively suitable, nevertheless it seems they approached their limit in this experiment as well.

Most suitable proved to be the DP 8005 adhesive, which proved great adhesion even to non-treated surface of low surface energy polyethylene, which determined in bond stronger than polyethylene itself. Nevertheless this adhesive is the most expensive one from all of the tested adhesives (see table 2.14 for comparison), therefore it should be considered whether to use this one without any need for surface treatment or another, cheaper, adhesive with plasma (or any other) treatment. For production of only a few bonded assemblies or home made products it may be better to use the DP 8005 adhesive. For industrial mass-production costs it may be more suitable to use a cheaper adhesive with surface plasma treatment.

As already mentioned in theoretical part of this work the EN 15337 standard declares, that "Design data may be obtained by testing of particular combination of adhesive and adherent material which is to be used in the actual assembly structure."[5] The fig 3.3 and fig 3.4 in attachment supports this statement. In the first figure shear test of five samples of non-treated PE bonded to aluminium using the DP 8005 are charted. In the second figure the blue curve in the bottom represents shear test of two non-treated polyethylene stripes bond together. Significant difference between bond strengths may be observed. Such difference in bond strengths may be caused by some process in adhesive initiated by contact with aluminium. In case of PE to aluminium bond it may have caused proper cross-linking of adhesive structure which led to stronger adhesive mass and adhesion failure between adhesive and polyethylene, whereas in case of PE to PE bond no such process occured, cured adhesive mass was weaker and therefore the cohesion failure occured. For future experiment I would recommend to examine bonding of various combinations of substrate using one adhesive and observe the influence of substrates on strength of adhesive mass and its structure.

For future work I would also recommend to test the six tested adhesives at samples with thicker polyethylene, in case of bonding polyethylene to aluminium, to ensure the PE substrate is not much weaker than adhesive in order to test the full potential of the adhesives.

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[22] 3M Company. *Epoxy Adhesive DP190 Grey Data Sheet*. St. Paul: Industrial Adhesives and Tapes Division, April, 2010.

[23] 3M Company. *Epoxy Adhesive DP100 Plus Clear Data Sheet*. St. Paul: Industrial Adhesives and Tapes Division, December, 2009.

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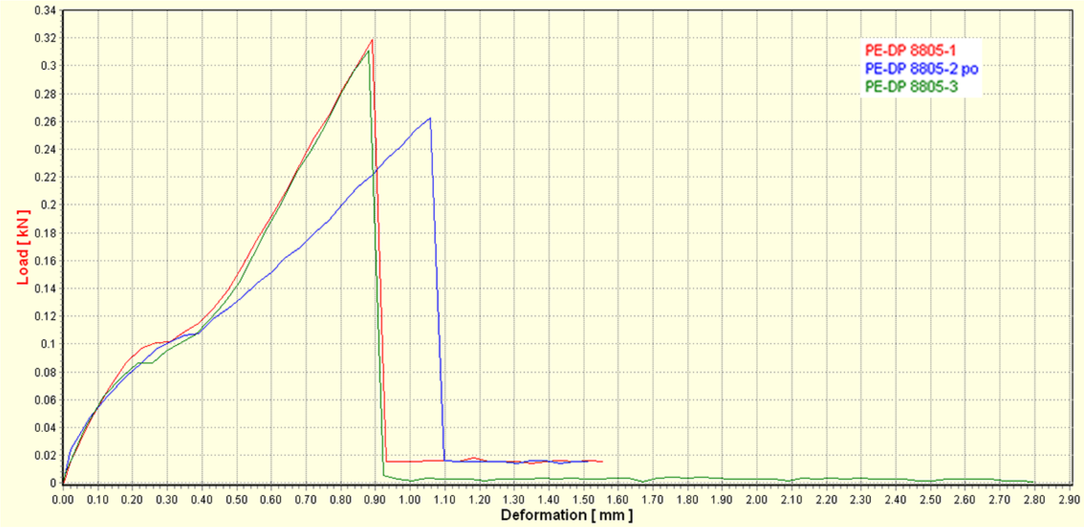
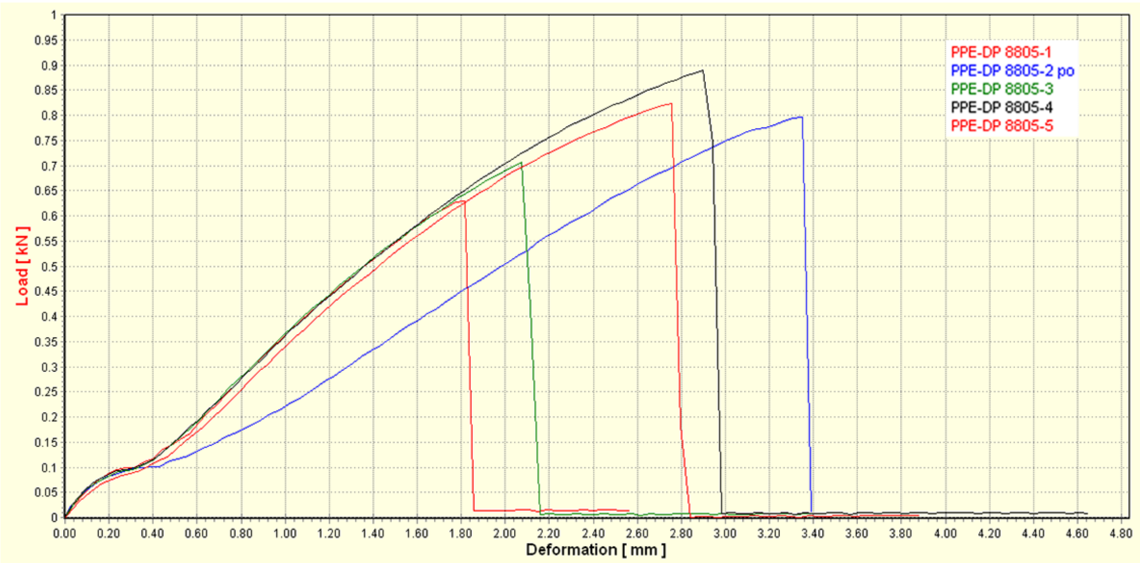


Figure 3.1: Chart of DP 8805 adhesive bond shear strength with PE tested according to EN 1465.



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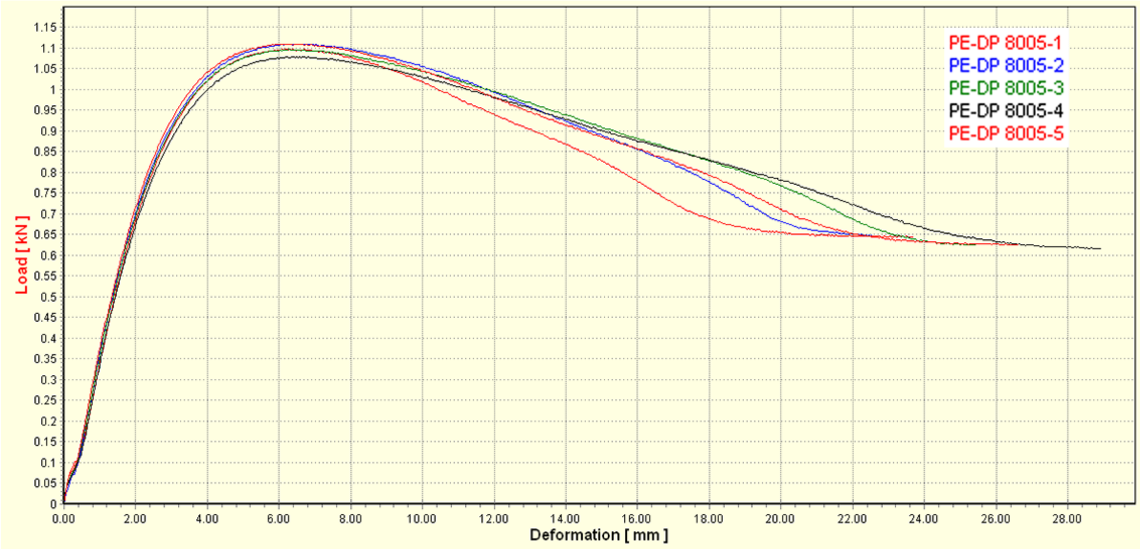

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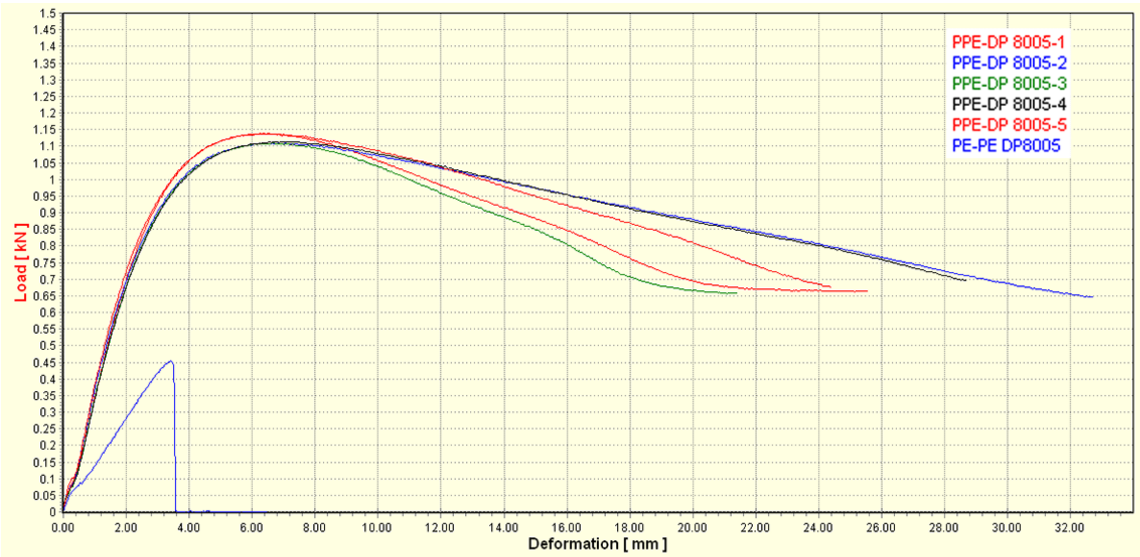

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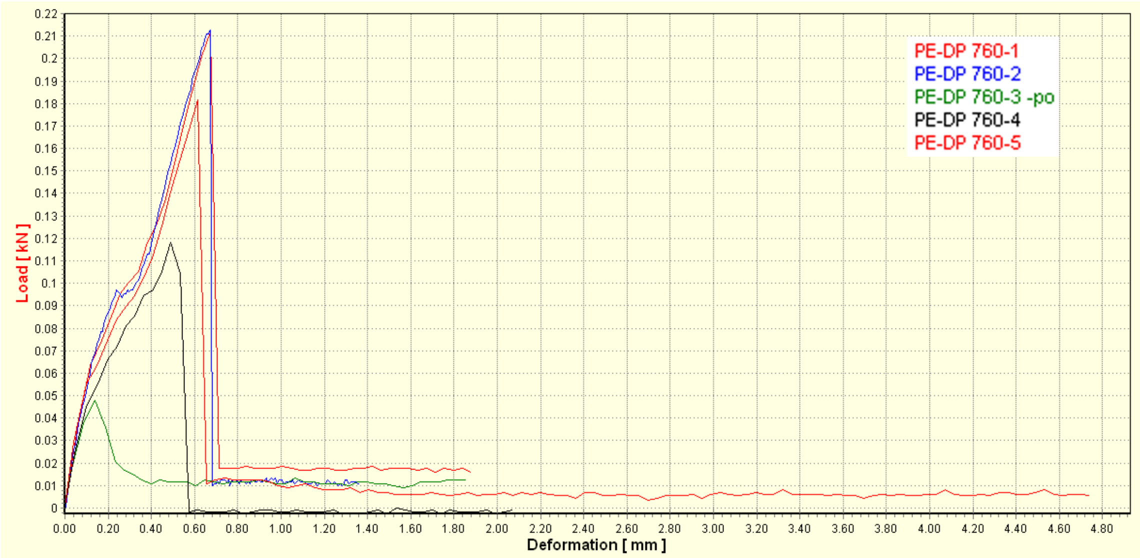

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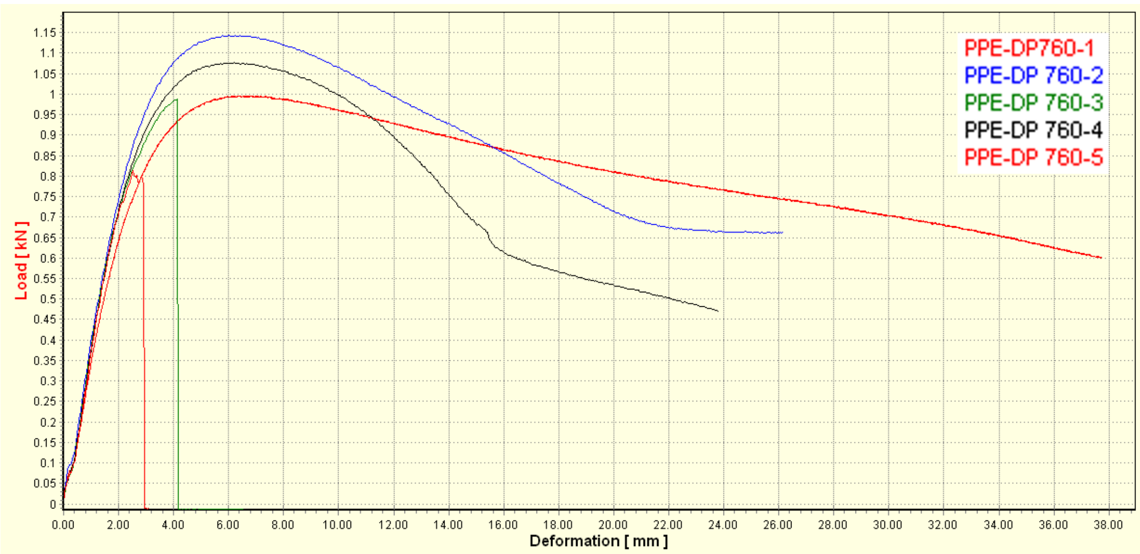

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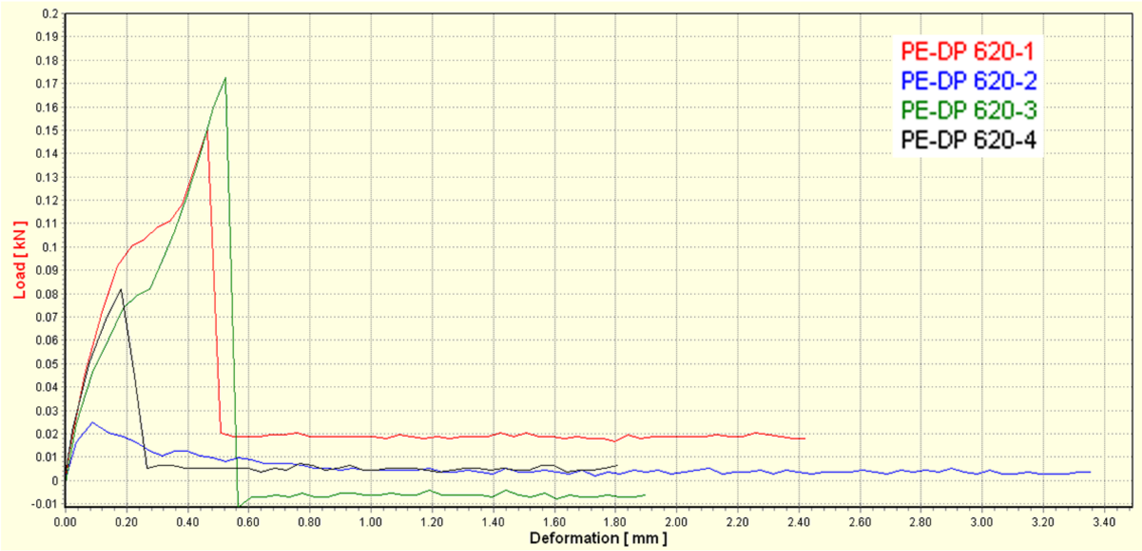

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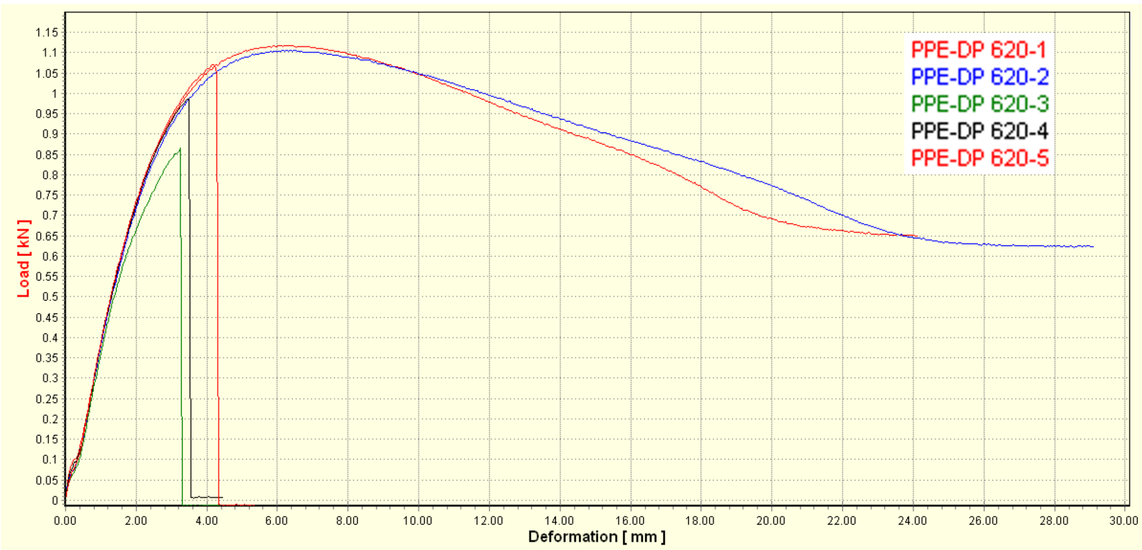

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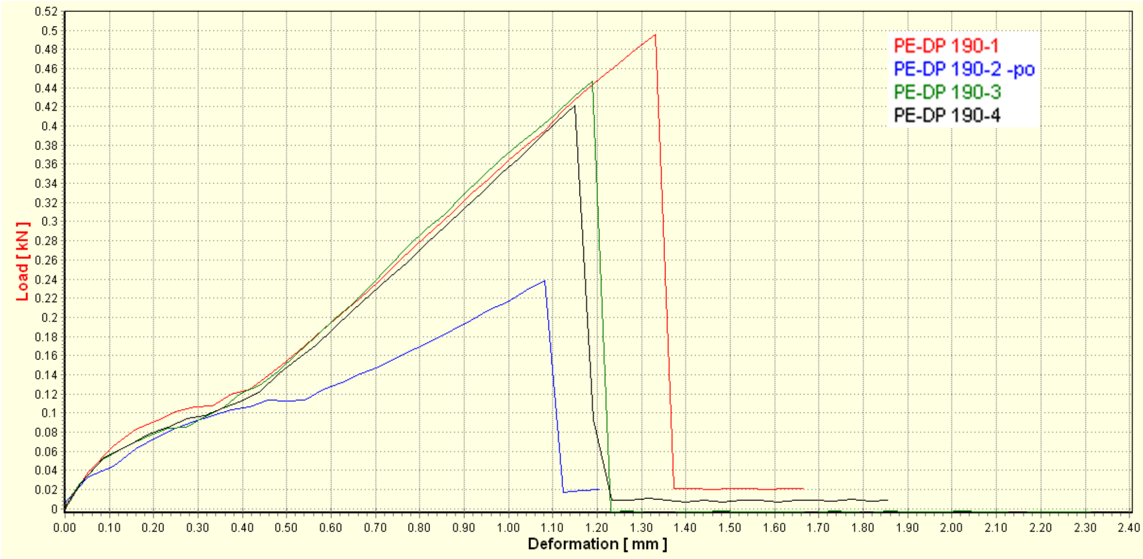

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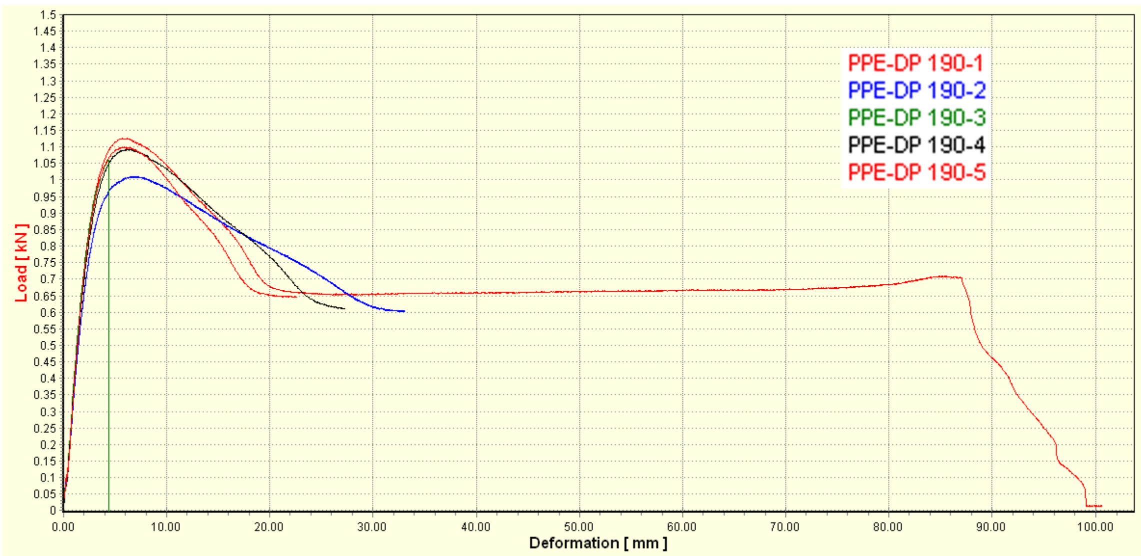

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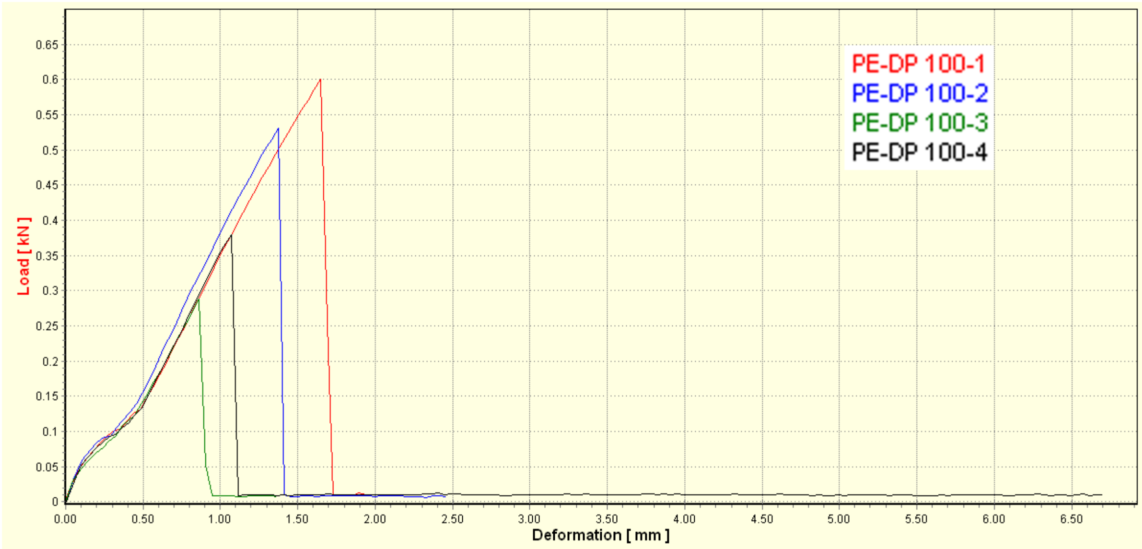

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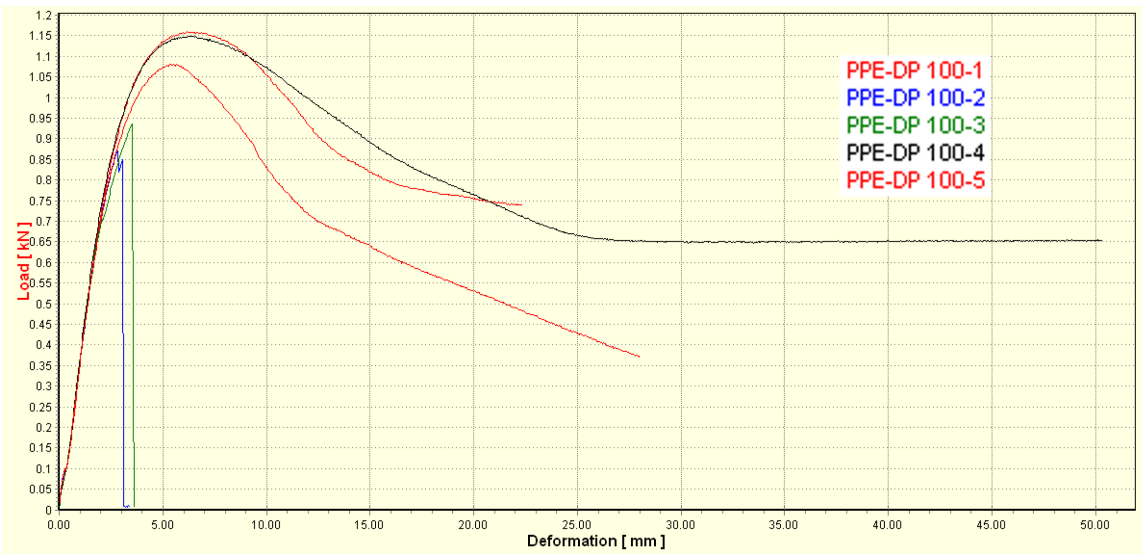

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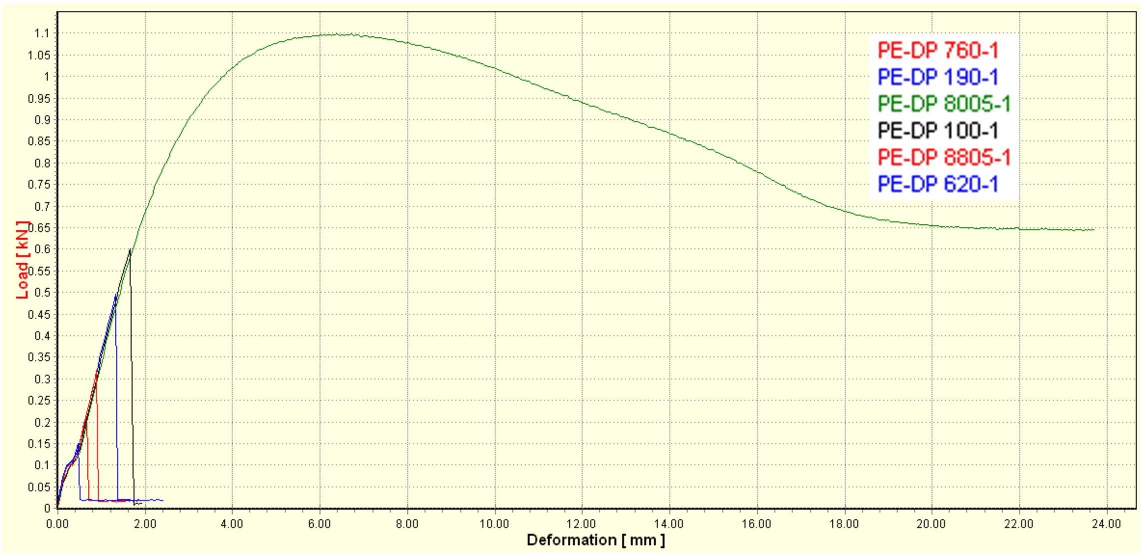

Figure 3.13: PE - measuring of influence of crosshead speed at material tensile strength