Available online at www.sciencedirect.com

ScienceDirect

Ciência & Tecnologia dos Materiais 27 (2015) 01–80

Ciência & Tecnologia dos MATERIAIS
http://ees.elsevier.com/ctmat

Special Issue on Ciência e Tecnologia dos Materiais

Analysis of polydicyclopentadiene-metal hybrid adhesive joints for automotive exterior body applications

Andre Camboa*, João Nunes[a], Fernando Alves[b]

[a] Institute of Polymers and Composites/I3N, Minho University, Campus de Azurém, 4800-058 Guimarães, Portugal
[b] INEGI/Faculty of Engineering, University of Porto, Rua Doutor Roberto Frias, 4200-465 Porto, Portugal

Abstract

An exterior body panel solution containing a polydicyclopentadiene skin attached to an interior metallic reinforcement through adhesive bonding is being studied to be applied in the MobiCar bonnet. With this solution is expected to achieve lightness, adequate structural integrity and cost-efficiency. However, there is uncertainty regarding to the bonnet adhesiveness since different metallic materials and adhesive types are being considered for its development. Thus, in this paper, several samples are tested through shear loading with the aim of understanding the loading magnitude expected by using polydicyclopentadiene, steel DC04+ZE and aluminum alloy AW5754-H111 as substrates adhesively bonded by an epoxy or a methacrylate.
Methacrylate adhesive have shown greater shear strength in all types of adhesive joints. PDCPD joints presented the highest displacements. Surface degradation was considered adequate over abrading once none strength difference was seen between the different surface treatments. Steel treated by cataphoresis has shown the highest joint interface strength.

Keywords: Polymer-metal hybrid joints; polydicyclopentadiene; aluminum; steel; adhesive;

1. Introduction

CEIIA (Portuguese Centre for Excellency and Innovation in Mobility Industry) is studying the development of an entire electric vehicle called MobiCar. With this vehicle, CEIIA aims to come up with enhanced technological and cost-efficient solutions on several areas, among them, powertrain, interior trims, seats and exterior body panels.

In the field of the exterior body panels, the polydicyclopentadiene (PDCPD) will be explored as exterior material. PDCPD presents good mechanical properties (30.0 kJ/m² of impact strength), good fracture and corrosion resistance, low density (1030kg/m^3) and good surface quality Class A, with already successful applications on agricultural machinery, trains and trucks [1-5]. PDCPD is conventionally processed through Reaction Injection Moulding (RIM), which allows polymer injection with low pressure (about 4MPa) and low temperature (<80º degrees Celsius) with production cycles that only takes few minutes, as well as reduced tooling investment. Such attributes, making it an attractive cost-efficient solution to manufacture small to medium series [6].

In order to explore this exterior solution, the MobiCar bonnet was developed by allowing lower complexity and prototyping costs while provides enough forecast accuracy regarding to engineering challenges. For the development of an adequate bonnet, a proper reinforcement structure, or inner

* Corresponding author. André Camboa, Engineering Desing and Advanced Manufaturing – Leaders for Technical Industries, MIT Portugal Program, Faculty of Engineering, University of Porto Porto, Portugal.
E-mail address: andre.camboa@fe.up.pt

panel has to be developed. Metallic materials such as steel or aluminum were considered to be attached to PDCPD since they provide enough stiffness to the entire element.

Steel offers easy of manufacturing with a wide range of possible application in the automotive industry. Depending on the steel grade, high deep drawing formability, adequate plasticity, good strength and stiffness properties as well as high impact absorbing capacity can be achieved. Besides that, with adequate dent resistance application and reduced thickness, lightweight results can also be achieved [7-9].

Aluminum presents low density (2750kg/m^3) and also excellent corrosion resistance [10]. Aluminum may also be assorted in several types of alloys that combining different percentages of additives results in different mechanical properties. 5xxx aluminum series are conventionally used in the interior panels due to its great deep drawing formability capacity [11].

The attachment of both exterior and interior panels was assumed to be accomplished through adhesive bonding. This process has already being used in the automotive industry and has proven to be effective on dissimilar materials, contributing for a good finishing and low stress concentration. No holes are required to execute the adhesion and it even allows the combination of thin and flexible adherents. Besides that, it is one of the most satisfactory joining methods when reduction of weight is required [12-22].

Considering this approach, the need of study these mentioned materials in what respect to its assemblability through adhesive bonding becomes evident. It aims looking for the differences of adhesiveness between steel, aluminum and DPCPD and the performance of several adhesives as well. It aims to overcome the lack of information regarding to adhesiveness of PDCPD. The study aims also to contribute, for instance, with information for the correct dimensioning of the glue track flap during the design stages of the components. Thus, this paper presents single lap shear tests performed over different materials previously thought for the MobiCar bonnet fabrication, which are introduced in the following topic.

1.1. Scope

Fig. 1 presents the configuration of the MobiCar bonnet which, was mentioned before, consisting in a polymer metal hybrid configuration (PMH) based in an exterior PDCPD element adhesively bonded through epoxy or methacrylate to an interior element that can be either in steel or aluminum.

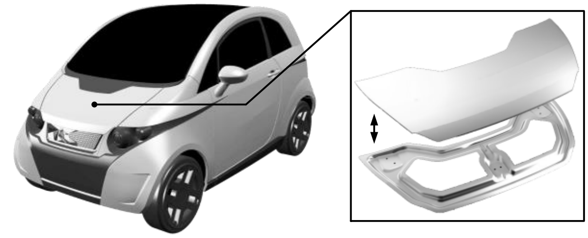

Fig. 1. MobiCar bonnet.

As PDCPD, Telene® 1650 series was used due to its balanced characteristics between rigidity and cycle times. As steel, the EN10152 DC04+ZE was used. It consists in a carbon steel coated by electrodeposition with a zinc layer enabling ensure good corrosion resistance. It also ensures adequate formability, often required for this type of automotive components fabrication. DC04+ZE composition can be seen through Table 1.

Table 1. EN10152 DC04+ZE chemical compound

Chemical element	%
Carbon (C)	0.08
Manganese (Mn)	0.40
Phosphoros (P)	0.03
Sulphur (S)	0.03

The steel element can be either in raw or in a treated condition, once it is known that steel parts that come from production often requires cleaning or degreasing or even surface treatment such as cataphoresis in order to promote adequate adhesiveness [23]. Considering this, samples of the DC04+ZE were also submitted to cataphoresis treatment in order to be compared to those non-treated.

As aluminum, the AW5754-H111 alloy was adopted and its chemical composition can be seen in Table 2. Besides the general benefits of aluminium, the AW5754-H111 alloy presents excellent corrosion resistance and formability. This alloy is specially indicated for vehicle bodies and shipbuilding.

No aluminum treated samples were considered for the study, once the surface finishing is apparently independently on the substrate material.

Table 2. AW5754-H111 chemical compound

Chemical element	%
Magnesium (Mg)	2.60-3.60
Manganese+Chromium (Mn+Cr)	0.10-0.60
Manganese (Mn)	0.0-0.50
Silicon (Si)	0.0-0.40
Iron (Fe)	0.0-0.40
Chromium (Cr)	0.0-0.30
Zinc (Zn)	0.0-0.20
Titanium (Ti)	0.0-0.15
Copper (Cu)	0.0-0.10

As adhesives, Araldite® 2015 and Crestabond® M7-15 were used as epoxy and methacrylate solutions, respectively. While epoxies guarantee the stiffest bond and are commonly used where/when higher loads are applied, methacrylate guarantee balanced properties of stiffness and ductility and is therefore appropriated when/where vibrations are involved [16, 22].

These adhesives suppliers present a diverse shear strength range for several materials, among them metals, composites and polymers which can be consulted elsewhere [24, 25]. However, for those metal grades defined before including PDCPD, there is no available data.

Considering this approach, the following diagram was built in order to show the bonnet configuration hypothesis and the samples configuration used within this study.

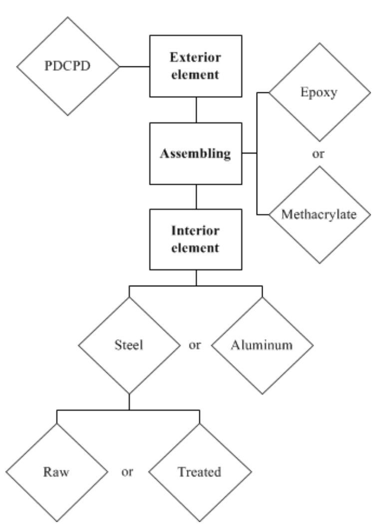

Fig. 2. Bonnet configuration hypothesis.

Once the measurement of subtracts adhesiveness is the main objective, all sample were fabricated with a mono-material configuration in order to avoid inaccurate measurements. By the end, only a sample containing a hybrid configuration was tested for results verification and discussion.

2. Experimental

The standards for determining shear strength of adhesively bonded rigid plastic (ASTM D 3163-01) and adhesively bonded metal specimens (ASTM D 1002-01) were reviewed. The results presented in this paper are expressed according to the average single lap joint shear stress formula expressed elsewhere [26]. Compensated substrates (Fig. 3 and Table 3), were fabricated in order to produce more accurate results by reducing the axial stress resulting from a non-uniform shear deformation.

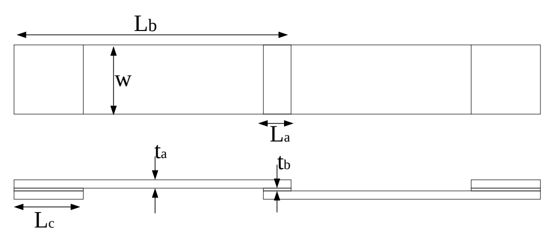

Fig. 3. Specimens geometry

Five different specimen's types were fabricated for each of four types of substrate materials: steel, painted steel, aluminum and PDCPD. Twenty five PDCPD subtracts were produced through milling from a rigid block with two different thicknesses: 7mm and 3mm. The thicker ones (7mm) were used to form a mono-material joint, and the thinners (3mm) were used for a hybrid joint. Forty subtracts of steel were cut from a sheet through guillotine cutting. Half of them were submitted to surface treatment. For the aluminum subtracts also twenty five were cut from a metal sheet by guillotine cutting.

Table 3. Adherent's geometrical dimensions

Quantity	Material	Dimensions					
		W	L_a	L_b	L_c	t_a	t_b
20	PDCPD	25	10	100	25	7	0.3
5	PDCPD	25	10	100	25	3	0.3
20	Steel	25	10	100	25	1.4	0.3
20	Steel+Cataphoresis	25	10	100	25	1.6	0.3
25	Aluminum	25	10	100	25	2	0.3

For almost all substrates a conventional manual abrasion through sand paper P100 with subsequent manual degreasing was applied. For PDCPD substrates was adopted a manual degreased with 96º ethylene alcohol while for metallic subtracts was adopted a manual degreasing with acetone. For steel painted and hybrid specimens only degreasing with 96º ethylene alcohol was adopted.

For the adhesive bond line thickness, 0.3mm was considered, which allows accomplishing the manufacturer recommendation of using a glue thickness between 0.1 mm and 1mm. Different curing times are advised by the adhesive manufacturers. The epoxy gel time is about 30 to 40 minutes while, methacrylate gel time is about 10 to 20 minutes considering a 1:1 mixing ratio. Also the fixture time is different, varying from 4 to 6 hours for epoxy and 30 to 45 min to methacrylate. The cure of all specimens was performed at room temperature for 5 days before being tested.

All the specimens were named based in its configuration in order to simplify data analysis. Their reference names are based in the first adherent material plus the adhesive plus the second adherent materials. The steel adherent is named with “ST”, and the steel submitted to cataphoresis paint is named as “KTL”. Polydicyclopentadiene is name as “DC” and aluminum as “AL5”. The adhesives are named by its manufacturer brand name, being Crestabond® M7- 15 named as “C715” and Araldite® 2015 as “A2015”. The specimens were tested at room temperature in an Universal testing machine Shimadzu AG-X-100KN, using a 100kN load cell with a span distance of 140mm between supports and 1mm/min speed.

2.1. Results

All the specimens presented coherent results regarding the type of failure obtained. Only one sample had failure on the substrate. A mixture of cohesive and adhesive failure was identified in whole mono-material aluminum samples. Table 4 shows the various type of failure achieved in the tests.

Table 4. Tensile test results (SF-substrate failure, CF- cohesive failure and AF- adhesive failure).

Reference	Specimen number				
	1	2	3	4	5
STA2015ST	AF	AF	AF	AF	AF
STC715ST	CF	CF	CF	CF	CF
KTLA2015KTL	CF	CF	AF	CF	CF
KTLC715KTL	CF	CF	CF	CF	CF
AL5A2015AL5	CF/AF	CF/AF	CF/AF	AF	CF
AL5C715AL5	CF/AF	CF/AF	CF/AF	CF/AF	CF/AF
DCA2015DC	CF	CF	CF	CF	CF
DCC715DC	CF	CF	CF	CF	CF
AL5C715DC	CF	CF	AF	SF	CF

Regarding to the whole sample of steel bonded with epoxy and methacrylate a variation from 15 to 10MPa may be seen. The epoxy itself presented strength values of about 10 to 14MPa and the methacrylate between 10 to 15MPa. Methacrylate presented larger deformations than the epoxy of about 0.5mm (Fig. 4 and 5).

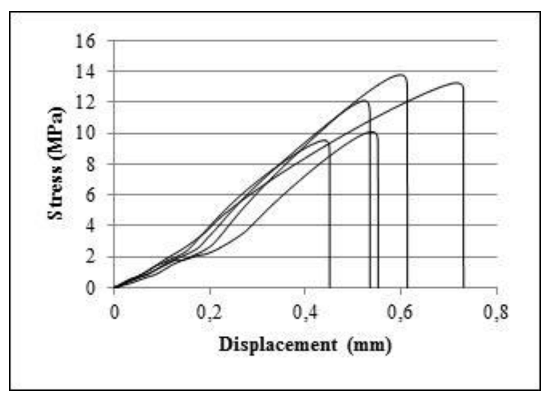

Fig. 4. Steel epoxy bonded results.

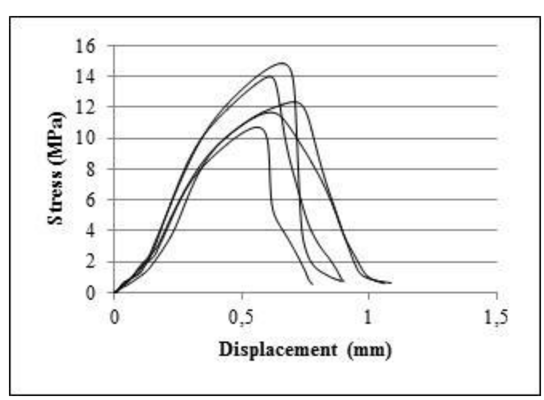

Fig. 5. Steel methacrylate bonded results.

The adherents submitted to the cataphoresis treatment shown the most increment in adhesive bond strength (Fig. 5 and 6). A maximum of 25MPa was obtained, which is the best case studied. Considering the epoxy adhesive, its values varied from 17 to 20MPa while, with the methacrylate the values varied from 20MPa to 25MPa. Globally, these results denoted substantial increment of strength when this treatment is applied on adherent parts. It can be concluded that, besides the need of steel surface treatment, cataphoresis is highly advised once it provides a substantial strength increment of about 40% comparing to steel not treated. However such increment requires an extra manufacturing stage and is only valid on its own adherent side if hybrid joints are produced.

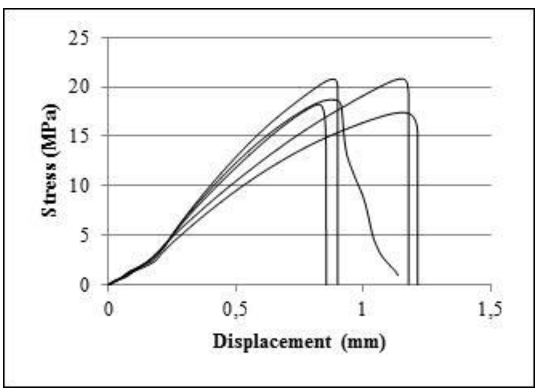

Fig. 6. Painted steel epoxy bonded results.

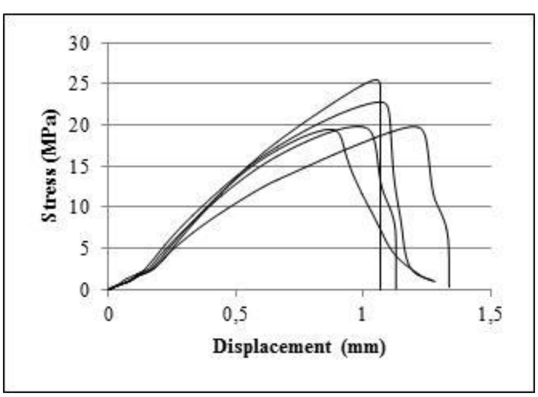

Fig. 7. Painted steel methacrylate bonded results.

Values between 14MPa and 16MPa of shear stress were achieved with the aluminum AW5754-H111 substrates (Fig. 8 and 9). It may be concluded that almost all displacement, about 1mm, was performed by the adhesive. The type of failure obtained is apparently a mix between cohesive failure, identified in the center of the joint, and adhesive failure at the joint edges.

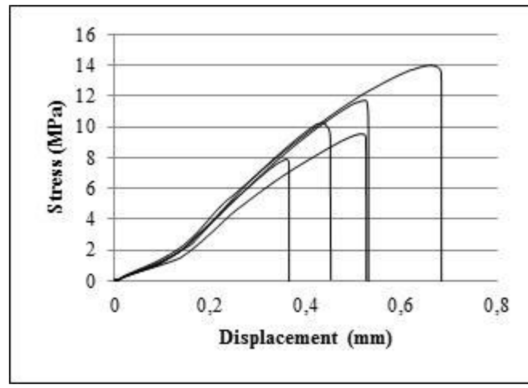

Fig. 8. Aluminum epoxy bonded results.

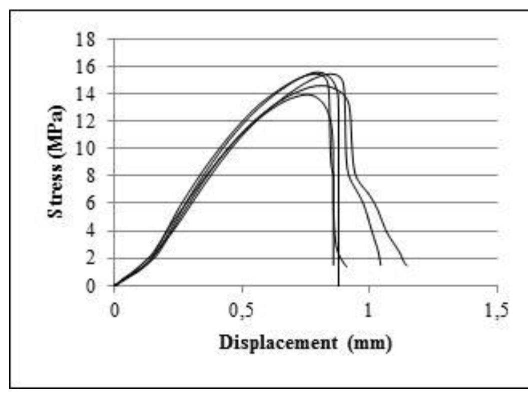

Fig. 9. Aluminum methacrylate bonded results.

A similar scenario was obtained by the PDCPD based adherents, but with inferior strength results. Whole PDCPD sample achieved values varying from 8 to 11MPa. Considering the results of PDCPD adherents glued with epoxy, a maximum value of 10MPa was achieved, while with methacrylate a maximum value of 11MPa was obtained (Fig. 10 and 11).

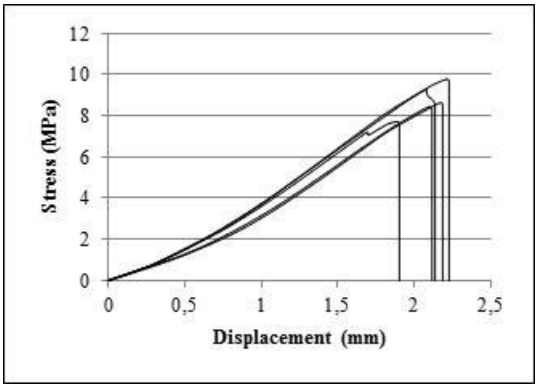

Fig. 10. PDCPD epoxy bonded results.

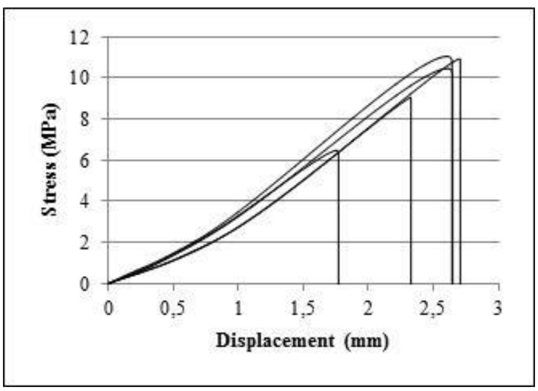

Fig. 11. PDCPD methacrylate bonded results.

Still regarding the mono material samples of PDCPD, a shear stress variation from 6MPa to 10MPa was obtained. However these specimens presented deformations of about 2.5mm, due to the lower Young Modulus of the substrates. In order to roughly calculate the elongation (δ) of the PDCPD substrates the following equation was applied:

$$\delta = FL/EA \quad (1)$$

where,
F is the maximum force recorded,
L is the length between grips accordingly to the Figure 3
E is the Young Modulus of PDCPD
A is the cross section area of the specimen according to Figure 3.

Considering that, the highest load value obtained was about 2500N, PDCPD has deformed 1.05mm from the 2.5mm depicted in Figure 11.

By the end, in order to simulate the bonnet configuration, hybrid specimens were fabricated. As it may be seen in Figure 12, the hybrid joints, composed by PDCPD and aluminum, presented shear stress values around 10MPa, which are similar to the mono-material PDCPD joints reported above through Figure 11. The displacement was about 2mm mainly due to the lower Young Modulus of the PDCPD as seen before. These types of joints presented three types of failure as may be seen in in the previous Table 4.

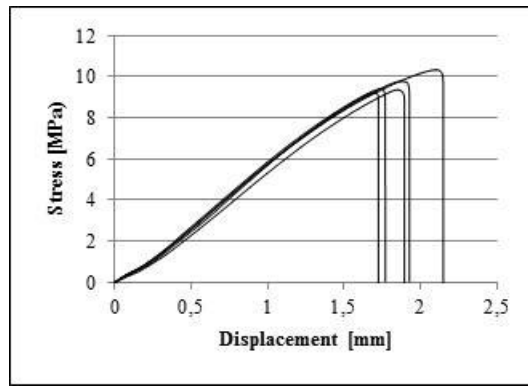

Fig. 12. Hybrid methacrylate bonded results

The PDCPD specimens presented the lowest shear strength results. It shows that the PDCPD surface presents poor interface properties. Considering that the mono-material PDCPD joints were abraded, contrarily to the hybrid solution, no significant difference in the joint strength was observed, it may be concluded that only degreasing is advisable.

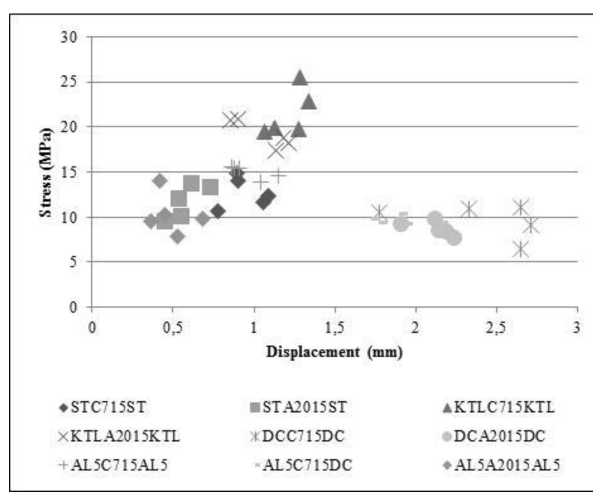

Fig. 13. Results overview.

2. Conclusions

From the study, several conclusions may be taken:

Methacrylate adhesive have shown to have greater shear strength (an average of 10%) in all types of adhesive joints comparatively to epoxy. Methacrylate presented also lower curing times.

Polydicyclopentadiene have shown the lowest interface strength results as well as the highest displacements due to its higher elasticity properties. Such ductility may be appreciated in parts were the fatigue cracking or vibrations must be controlled.

Considering the mono-material PDCPD joints were abraded, contrarily to the hybrid solution, and no significant difference in the joint strength was observed, it may be concluded that only degreasing is advisable.

Raw steel and aluminum presented similar results. Cataphoresis treatment is highly advised since an increment of strength of about 40% was achieved. However such increment requires an extra manufacturing stage and production costs which may vary accordingly to each part geometry.

Acknowledgements

This work is financed by FEDER Funds, through the Operational Programme for Competitiveness Factors - COMPETE under the project 13844.

André Camboa thanks FCT (Fundação para a Ciência e Tecnologia) for PhD scholarship (SFRH / BD / 51119 / 2010) under the scope of the MIT Portugal Program in Engineering Design and Advanced Manufacturing - Leaders for Technical Industries focus area.

References

[1] Y. Weijun Yin Weijun, S. Kniajanski, B. Amm, Ieee International Symposium On Electrical Insulation. Proceedings, (2010) 1-5.
[2] M.J. Abadie, M. Dimonie, C. Couve, V. Dragutan, European Polymer Journal, 36 (2000) 1213-1219.
[3] S.E. Barnes, E.C. Brown, N. Corrigan, P.D. Coates, E. Harkin-Jones, H.G. Edwards, Spectrochimica acta. Part A, Molecular and biomolecular spectroscopy, 61 (2005) 2946-2952.
[4] A. Teixeira, B. Ribeiro, Rapid Product Development Event, (2010).
[5] V.J. Toplosky, R.P. Walsh, AIP Conference Proceedings, 824 (2006) 219-224.
[6] G.S. Constable, A.J. Lesser, E.B. Coughlin, Macromolecules, 37 (2004) 1276-1282.
[7] K.L. Edwards, Materials & Design, 25 (2004) 529-533.
[8] Y. Zhang, X. Lai, P. Zhu, W. Wang, Materials & Design, 27 (2006) 64-68.
[9] A.M. Sherman, A. Krause, P. Friedman, D.Q. Huston, Automotive Body Materials, in: K.H.J.B. Editors-in-Chief: , W.C. Robert, C.F. Merton, I. Bernard, J.K. Edward, M. Subhash, V. Patrick (Eds.) Encyclopedia of Materials: Science and Technology (Second Edition), Elsevier, Oxford, 2001, pp. 415-421.
[10] T.A. Association, The Aluminum Association, 1998.
[11] W.S. Miller, L. Zhuang, J. Bottema, A.J. Wittebrood, P. De Smet, A. Haszler, A. Vieregge, Materials Science and Engineering: A, 280 (2000) 37-49.
[12] S.T. Amancio-Filho, J.F. dos Santos, Polymer Engineering & Science, 49 (2009) 1461-1476.
[13] J. Rotheiser, Joining of Plastics 3E: Handbook for Designers and Engineers, Hanser Gardner Publications, 2009.
[14] J.-H. Kweon, J.-W. Jung, T.-H. Kim, J.-H. Choi, D.-H. Kim, Composite Structures, 75 (2006) 192-198.
[15] P. Davies, L. Sohier, J.Y. Cognard, A. Bourmaud, D. Choqueuse, E. Rinnert, R. Créac'hcadec, International Journal of Adhesion and Adhesives, 29 (2009) 724-736.
[16] P. Feraboli, A. Masini, Composites Part B: Engineering, 35 (2004) 323-330.
[17] T.A. Barnes, I.R. Pashby, Journal of Materials Processing Technology, 99 (2000) 72-79.
[18] L.D.R. Grant, R.D. Adams, L.F.M. da Silva, International Journal of Adhesion and Adhesives, 29 (2009) 535-542.
[19] L.D.R. Grant, R.D. Adams, L.F.M. da Silva, International Journal of Adhesion and Adhesives, 29 (2009) 405-413.
[20] A. Beevers, S.M. Steidler, J. Durodola, M. Coackley, Journal of Materials Processing Technology, 118 (2001) 95-100.
[21] A. Higgins, International Journal of Adhesion and Adhesives, 20 (2000) 367-376.
[22] D.E. Packham, International Journal of Adhesion and Adhesives, 29 (2009) 248-252.
[23] E. Almeida, I. Alves, C. Brites, L. Fedrizzi, Progress in Organic Coatings, 46 (2003) 8-20.
[24] Huntsman, in: Huntsman (Ed.).
[25] S.B. Adhesives, in: S.B.C. LIMITED (Ed.), www.scottbader.com, 2014.

[26] R.D. Adams, J. Comyn, W.C. Wake, Structural Adhesive Joints in Engineering, Springer, 1997.