

HIGH TEMPERATURE IMPACT ASSESSMENT ON THE SHEAR STRENGTH OF ADHESIVELY BONDED JOINTS IN MULTI-MATERIAL DESIGN

Paweł ZDZIEBKO*, Jakub KORTA, Tadeusz UHL

AGH University of Science and Technology
Faculty of Mechanical Engineering and Robotics
Department of Robotics and Mechatronics
al. A. Mickiewicza 30, 30-059, Kraków, Poland
**zdziebko@agh.edu.pl*

Summary

Multi-material joints are implemented in many fields, including automotive and aerospace industries. Adhesive bonding technique allows for joining components made of different materials, which facilitates the implementation of lightweight materials in multi-material structures. In this study the shear strength of single lap, adhesively bonded joints was examined at room and high temperature (110°C). In the paper the influence of elevated temperature on epoxy adhesive joints in multi-materials structures is presented. A sensitivity of shear strength on material type of adherents in adhesively bonded joints is considered. Prediction of the state of joints and knowledge on their degradation process are important for structural health monitoring of structures with adhesive joints.

For the purposes of research, specimens consisting of adherents made of various materials were used (following were involved: aluminum alloy, CFRP and abrasive resistant steel). Studies have demonstrated, that applied epoxy adhesive considerably weakens at elevated temperature and it was observed that material type of adherents has a large influence on the shear strength of adhesively bonded joints.

Keywords: glues, adhesives, bonding, high temperature, multi-material joints.

OCENA WPŁYWU PODWYŻSZONEJ TEMPERATURY NA WYTRZYMAŁOŚĆ NA ŚCINANIE POŁĄCZEŃ KLEJONYCH W KONSTRUKCJACH WIELOMATERIAŁOWYCH

Streszczenie

W wielu gałęziach przemysłu, włączając sektor samochodowy oraz lotniczy, obserwuje się potrzebę łączenia ze sobą elementów wykonanych z różnych materiałów. Metodą pozwalającą spełnić to wymaganie jest klejenie adhezyjne. Prezentowane poniżej badania eksperymentalne wytrzymałości na ścinanie przeprowadzono w temperaturze pokojowej oraz w temperaturze wysokiej (110°C). W artykule opisano wpływ oddziaływania wysokiej temperatury na połączenia klejone w konstrukcjach wielomateriałowych, rozważa się wpływ kombinacji klejonych materiałów na wytrzymałość połączeń. Przewidywanie stanu złącza oraz wiedza na temat procesu jego degradacji niosą istotną informację z punktu widzenia monitorowania stanu konstrukcji klejonych. Przebadało próbki klejone jednozakładkowe, zastosowano elementy składowe próbek wykonane z różnorodnych materiałów (wykorzystano stop aluminium, kompozyt węglowy jednokierunkowy oraz stal trudnościeralną). Badania wykazały, że badany klej epoksydowy ulega znacznemu osłabieniu w podwyższonej temperaturze, ponadto zaobserwowano istotny wpływ rodzaju klejonych materiałów na wytrzymałość próbek.

Słowa kluczowe: kleje, adhezja, klejenie, wysoka temperatura, złącza wielomateriałowe.

1. INTRODUCTION

Adhesive bonding is one of the techniques of creating inseparable mechanical joints, it consists of creating a permanent connection by disposing a glue layer between the parts. Bonded elements are fastened together due to the adhesion forces, which are the intermolecular interaction between adherent

and glue layer surfaces, and cohesion forces, which arise from internal consistency of the glue layer [1].

Adhesively bonded joints usually enable to reduce weight and complexity of structure comparing with classical material joining techniques like bolting and riveting by reducing the number of important components to create the joint. The adhesive bonding has a very good strength to weight ratio due to the small unit mass in comparison with

conventional joining methods and has high efficiency because of its simplicity [2]. The most important benefit of using bonding technology is the ability to join adherents made of various materials, which is impossible to achieve when using other joining methods, like spot welding technique. Additionally, this method does not distort the components being joined and also do not introduce initial stress in the joint. These advantages of the adhesive bonding method are particularly important from the point of view of the current trends in the automotive sector, which are focused on decreasing the structural mass of a vehicle, by implementing lightweight materials in car body structure, without loss in its mechanical properties as described by Korta and Uhl [3]. The glue layer might also protect joints against galvanic corrosion, because it separates the adherents. On the other hand, the adhesive bonding technique has several disadvantages and limitations. One of the major drawbacks is that bonded joints might be responsive to varying temperature and other environmental conditions. Many of the structural adhesives require heat curing, which might introduce complications to the assembly process. It is also considered that the adhesive joints are inherently weak in peel [4].

With the increased use of modern structural materials in the engineering, the subject of adhesively bonded joints is gaining interest of many scientists. Research on adhesively bonded joints frequently relate to connections burdened with shear load [5]. Godzinski's [6] work presented important aspects according to the adhesive bonding technology. The author described and presented test results of structural limitations, static durability and fatigue strength of the bonded joints. Wahab [7] discussed fatigue properties of this type of connections. Many researchers investigate the influence of environmental conditions, varying temperatures or surface treatment on the strength of bonded joints, the most often considering one type of adherent combination. Hu et al. [8] discussed in their studies the effects of long-term wide range temperature exposure on the single lap joints comprising of steel and aluminum adherents. Researchers indicated that long-term high or low temperature exposure weakens the bonded joint strength. Silva and Adams [9] studied dual adhesives joints to be used over a wide temperature range (-55°C – 200°C). Authors have demonstrated that for the joints with dissimilar materials, the combination of two adhesives gives better results across the investigated range of temperatures than a high temperature resistant adhesive alone. Multi-material bonded joints were also investigated by other authors: Raykhere et al. [10] and Kang et al. [11]. Kim et al. [12] examined the durability of the double lap shear joints subjected to the typical cold region conditions. The viscoelasticity of the adhesives was discussed by Rośkowicz [13]. Rudawska [14] have shown in her study how the adherents surface treatment can influence the

epoxy-based joints strength. Research undertaken by her was carried out on specimens made of stainless steel. Also Kłonica and Kuczmazewski [15] have shown in their research that the surface preparation has a large effect on the strength of adhesively bonded elements made of aluminum alloy.

Many authors focus on the methods for Non-Destructive Testing (NDT) and Structure Health Monitoring (SHM) of bonded joints, which allows for constant and *in situ* assessment of adhesive joints condition without disturbing the connection, what is very significant in critical structures. Authors use different approaches including guided waves [16], optical fiber sensors [17] and others like vibrothermography and ultrasonic A-Scan and C-Scan. Staszewski and Dao [18] [19] emphasize the impact of temperature variations on SHM methods. Operating temperature may also has influence on the ultimate strength of adhesive joints, thus strength investigation in various conditions is necessary in terms of structure health monitoring of adhesive joints. The following studies are the basis for further research on SHM methods.

The goal of the authors work is to design the SHM method which is dedicated to predict the health of adhesive joints. The idea of the method contains measurements of environmental and load conditions and based on the model of degradation assessment of health of the joints. Presented studies are focused on experimental testing of adhesive joints condition for particular type of a glue which are the basis of the method. The paper is organized in the following way; chapter 2. contains specification of conditions of conducted tests, chapter 3. describes test specimens, chapter 4. presents the results obtained at experiment, chapter 5. includes a summary and conclusions.

2. DESCRIPTION OF THE TESTS

To ensure the safety of adhesively bonded joints, it is necessary to analyze its strength at its working conditions. This paper treats the subject of the shear strength investigation of the adhesively bonded joints made of dissimilar materials subjected to the impact of high temperature. The selection of test conditions and materials types of the test specimens refers to the structure of automotive vehicle, in which the manufacturer considers the implementation of adhesively bonded joints. The temperature around 110°C was considered to be the maximum that may occur in the bonded joint area for a short time. Determination of the joint's ultimate strength at elevated temperature is crucial in the assessment of joint behavior even if the durability of the joint at elevated temperature may be significantly lower than in its normal working conditions.

The goal of this experiment is to explore:

- influence of operation condition on quality of adhesive joints,

- if the material type combination of adherents has influence on the shear strength of the bonded joints,
- if the temperature of $110^{\circ}\pm 2^{\circ}\text{C}$ significantly decreases the ultimate shear strength of joints prepared using Loctite Hysol®9497 adhesive,
- which adherents combination is recommended for adhesive joint preparation with respect to operating at high temperature.

The samples were divided into two groups, one was tested at the room temperature (RT) and the second one was tested at the temperature of $110^{\circ}\pm 2^{\circ}\text{C}$ (HT), to examine the effect of high temperature on the multi-material, adhesively bonded joints.

3. TEST SPECIMENS

The two-component, epoxy adhesive Loctite Hysol®9497 was used for the test specimen preparation. This adhesive is widely available and has good and uniform strength parameters at elevated temperature eg. in automotive structures. It was decided to use three types of joined materials: aluminum alloy EN AW 5083 (ALU), unidirectional carbon fiber reinforced polymer (CFRP) and Hardox Extreme® steel - an abrasion resistant steel (HARDOX), which are commonly used engineering. The parameters describing these materials are presented in Tab. 1. Samples of the following adherent combinations were prepared (in required amount):

- ALU – ALU,
- CFRP – CFRP,
- ALU – CFRP,
- ALU – HARDOX,
- CFRP – HARDOX.

Tab. 1. Mechanical properties of used materials

Material	Young's modulus [GPa]	Poisson ratio	Density [g/cm ³]	Tensile strength [MPa]
ALU	71.4	0.33	2.65	300
HARDOX	210.0	0.3	7.89	1250
CFRP 0°/90°	91.7/4.9	0.31 (ν_{12})	1.65	1250/40
Hysol® 9497	2.4	0.3	2.02	52.6

The test specimen geometry (Fig. 1) and whole tests were carried out according to the ASTM D3165 test method. Thickness reduction from 4 to 2 mm was achieved by mechanical treatment for

ALU and HARDOX components and by bonding two strips of different lengths for CFRP elements.

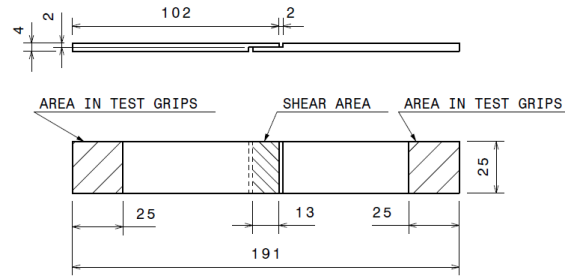


Fig. 1. The form and dimensions of the test specimens

For each substrates combination, the amount of 20 patterns were tested for the individual research conditions. Before bonding, components were degreased – in case of components made of HARDOX and CFRP, or treated by abrasive paper and then cleaned and degreased – in case of components made of aluminum alloy. After preparation, the test specimens were conditioned at room conditions for at least 14 days and then tested on Instron 8872 testing machine with CP100557 environmental chamber. The quasi-static load was applied with the velocity of 1.27mm/min. The test stand and exemplary prepared specimen mounted in the test machine holders is presented in Fig. 2.

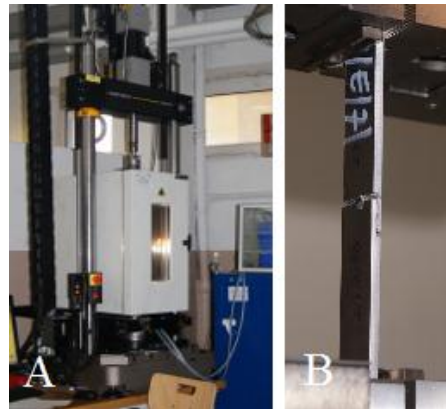


Fig. 2. A – Test machine and environmental chamber, B – Exemplary specimen mounted in test machine holders

4. RESULTS

The shear strength tests of the bonded joints were conducted at room temperature and at the temperature of $110^{\circ}\pm 2^{\circ}\text{C}$ (after heating up the chamber for at least 1.5 hour and the samples for at least 15 minute). The most important parameter obtained in the tests was tension at failure of bonded joint (Tab. 2, Fig. 6). Following parameters were used for a statistical description of the results:

Mean value of shear strength (1):

$$\overline{Rm} = \frac{1}{n} \sum_{i=1}^n Rm_i \quad (1)$$

Standard deviation of shear strength:

$$\sigma = \sqrt{\frac{\sum_{i=1}^n (Rm_i - \overline{Rm})^2}{n}} \quad (2)$$

Strength loss at the temperature of $110^{\circ}\pm 2^{\circ}\text{C}$:

$$\Delta R_m = \frac{(\overline{Rm}_{RT} - \overline{Rm}_{110^{\circ}}) \cdot 100\%}{\overline{Rm}_{RT}} \quad (3)$$

Where:

n – number of tested specimens,

Rm_i – shear strength value obtained for a single specimen

Tab. 2. The shear strength test results

Specimen type	Test at room temperature		Test at $110^{\circ}\pm 2^{\circ}\text{C}$		ΔR_m [%]
	\overline{Rm}_{RT} [MPa]	σ_{RT} [MPa]	$\overline{Rm}_{110^{\circ}}$ [MPa]	$\sigma_{110^{\circ}}$ [MPa]	
CFRP – CFRP	14.29	1.73	9.24	1.44	35.3
CRFP – HARD OX	8.28	1.72	3.35	1.6	59.5
CFRP – ALU	7.0	2.50	6.42	2.76	8.3
ALU – ALU	8.61	1.77	8.3	2.01	3.6
ALU – HARD OX	9.98	2.09	6.99	2.81	30.0

In addition to the shear strength results, also the way of the joints fracture brings relevant information about quality of the joints preparation. In examined groups of specimens, four typical fracture types were observed: cohesive fracture, adhesive fracture, fracture at material layer and mixed fracture (Fig. 3-Fig. 5).



Fig. 3. The CFRP-CFRP specimen tested at RT, the material layer failure (fibers torn from the surface)



Fig. 4. The CFRP-HARDOX specimen tested at RT, the adhesive failure (adhesive remains on one adherent)



Fig. 5. The CFRP-ALU specimen tested at HT, the cohesive failure (adhesive remains on both adherents)

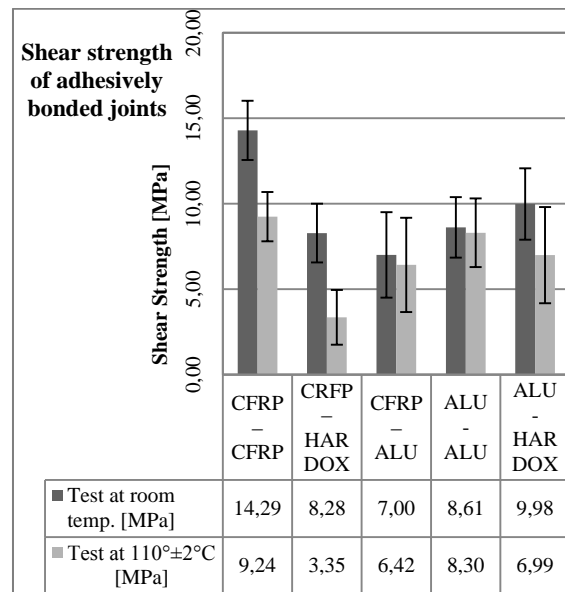


Fig. 6. The shear strength of bonded joints

Generalized fracture observations for all groups of specimens are presented in Tab. 3.

Tab. 3. Typical mode of the specimens fracture

Specimens type	Test at RT	Test at HT
CFRP-CFRP	material	mixed
CFRP-HARDOX	adhesivly from HARDOX	adhesivly from HARDOX
CFRP-ALU	adhesivly from ALU	cohesive
ALU-ALU	adhesivly from both	cohesive
ALU-HARDOX	adhesivly from both	adhesivly from HARDOX

The groups of patterns which contained elements made of HARDOX steel, the most frequently crack with adhesive fracture from steel surface. This fact shows that the way of preparation HARDOX steel surface before bonding was insufficiently adequate (HARDOX bonded surface was very smooth) or a composition of HARDOX steel adversely affects the bonded joint strength (HARDOX steel contains 1.2% chromium which negatively influences the adhesive properties). It was observed that samples containing adherents made of CFRP (which surface is rugged) very rarely crack with adhesive fracture from CFRP surface, which indicates good properties of adopted composite material for adhesive bonding process. Although aluminum adherents were treated by abrasive paper as the only material, crack with adhesive failure from this substrate was frequently observed.

In the case of the tests at room temperature, CFRP-CFRP samples have the biggest shear strength among all patterns groups (14,29 MPa). Moreover, low standard deviation of results was noted (1,73MPa) for this group. Material layer fracture of CFRP-CFRP samples indicates a high shear strength of this type of bonded joints, which is higher than material strength. These observations of mentioned test group lead to the conclusion, that CFRP material has good adhesive properties – this fact might be caused by high roughness of this material (which was produced in pultrusion process). The second largest shear strength obtained at RT was noted for ALU-HARDOX samples (9,98MPa). CFRP-ALU is the only test group which achieved the shear strength below 8 MPa at the test conducted at room temperature. Adhesive fracture observed for CFRP-ALU specimens indicates not sufficiently proper ALU surface treatment before bonding process, what can be additionally provided by high standard deviation for the results for this test group. Similar values of the shear strength were noted for CFRP-HARDOX and ALU-ALU specimens, respectively 8,28 MPa and 8,61 MPa.

Analyzing the results obtained at the temperature of 110°C, the coefficient of thermal expansion (CTE) of materials is an important factor, which may have large influence on the strength of specimens tested at HT.

Significant thermal expansion differences (Tab. 4.) of materials and adhesive layer used for producing patterns might introduce additional stress at the joint area under the influence of temperature variation. This observation is supported by numerical analysis carried out by Korta [22].

The highest shear strength values at HT tests were noted for joints consisting of adherents made of the same material type: CFRP-CFRP (9,24 MPa) and ALU-ALU (8,3 MPa), which have the same coefficient of thermal expansion for both of adherents. On the other hand, CFRP-CFRP test group (which at RT also has the largest shear strength

among all types of specimens) exhibits large shear strength reduction at HT (35.30%). For CFRP-CFRP samples tested at HT, the lowest standard deviation of shear strength results was noted (1,44MPa), which proves the high repeatability of the results for this test group. Taking into account changes at the way of fracture for this type of specimens, arguments brought forward clearly show that Loctite Hysol®9497 significantly weakens at the temperature of 110°C. ALU-ALU and CFRP-ALU samples have the lowest loss of strength caused by high temperature impact among all other test patterns, respectively 3,60% and 8,30%.

Tab. 4. Thermal expansion coefficients for the tested materials.

Material type	Coefficient of thermal expansion [$10^{-6}/^{\circ}\text{C}$]
Loctite Hysol® 9497	104 [5]
ALU	22.3-26 [20]
CFRP (unidirectional)	(-1) – 2 (longitudinal), 22-47 (transverse) [21]
HARDOX	9.4 - 15 [20]

For the test specimens types containing adherents made of HARDOX (CFRP-HARDOX and ALU-HARDOX) large reduction of shear strength was noted. The highest loss of strength due to the high temperature influence among all test groups was observed for CFRP-HARDOX specimens – 59,5%, in the case of ALU-HARDOX samples it was 30,00%.

5. CONCLUSIONS

The goal of this research was to specify changes in condition of adhesively bonded joints at elevated temperature (112°C), the results are the basis of the SHM method which is constantly developed by the authors.

The studies have shown the reduction in shear strength of the bonded joints (made using Loctite Hysol®9497epoxy adhesive) at high temperature, regardless of the material combination of adherents. The largest shear strength reduction under the influence of high temperature was noted for CFRP-HARDOX specimens and this type of joints is not recommended to operate at elevated temperature. The shear strength above 6MPa at HT was noted for other types of materials combinations - CFRP-CFRP, CFRP-ALU, ALU-ALU, ALU-HARDOX, and there is a possibility to implement them in automotive vehicle structure operating at elevated temperature for a short time. For these specimens

types the shear strength above 6MPa was noted also at the test at RT.

From the point of view of assessing the state of bonded joints in multi-material structures, the influence of operating conditions on state of adhesive joints is relatively large. Conclusion drawn from the research is that, to assess actual state of adhesive joints the operation conditions should be monitored. The humidity, temperature and loads are most important factors which influence properties of joints. Change in working conditions may significantly decrease the strength of adhesively bonded joints. The following results are motivation for further investigation on structure health monitoring of bonded joints.

The mean values of shear strength obtained at this research indicate large potential for joining dissimilar materials using adhesive bonding technology. On the other hand, large standard deviation (noted for the most of the samples) shows difficulties in creating adhesively bonded joints with repeatable shear strength. Moreover, recorded results prove the importance of the surface preparation in the adhesive bonding process.

REFERENCES

- [1] Rudawska A., *Wybrane zagadnienia konstruowania połączeń adhezyjnych jednorodnych i hybrydowych*, Lublin: Politechnika Lubelska, 2013.
- [2] Sayman O., *Elasto-plastic stress analysis in an adhesively bonded single-lap joint*, Composites: Part B, no. 43, pp. 204-209, 2012.
- [3] Korta J. and Uhl T., *Multi-material design optimization of a bus body structure*, Journal of KONES : Powertrain and Transport, vol. 20, no. 1, pp. 139-146, 2013.
- [4] Barnes T.A. and Pashby I.R., *Joining techniques for aluminium spaceframes used in automobiles Part II - adhesive bonding and mechanical fasteners*, Journal of Materials Processing Technology 99, pp. 72-79, 2000.
- [5] Rudawska A., *Wybrane zagadnienia konstruowania połączeń adhezyjnych jednorodnych i hybrydowych*, Lublin: Politechnika Lubelska, 2013.
- [6] Godzimski J., *Problemy klejenia konstrukcyjnego*, Technologia i Automatykacja Montażu, no. 1, pp. 25-31, 2009.
- [7] Wahab M.M.A., *Fatigue in Adhesively Bonded Joints: A Review*, International Scholarly Research Network, 2012.
- [8] Hu P., Han X., Li W.D., Li L. and Shao Q., *Research on the static strength performance of adhesive single lap joints subjected to extreme temperature environment for automotive industry*, International Journal of Adhesion & Adhesives 41, p. 119–126, 2013.
- [9] Da Silva L.F.M. and Adams R.D., *Adhesive joints at high and low temperatures using similar and dissimilar adherends and dual adhesives*, Adhesion & Adhesives 27, p. 216–226, 2007.
- [10] Raykhere S.L., Kumar P., Singh R.K. and Parameswaran V., *Dynamic shear strength of adhesive joints made of metallic and composite adherents*, Materials and Design 31, pp. 2102-2109, 2010.
- [11] Kang S.G., Kim M.G., Kim C.G., *Evaluation of cryogenic performance of adhesives using composite–aluminum double-lap joints*, Composite Structures 78, pp. 440-446, 2007.
- [12] Kim Y.J., Hossain M. and Yoshitake I., *Cold region durability of a two-part epoxy adhesive in double-lap shear joints: Experiment and model development*, Construction and Building Materials, no. 36, 2012.
- [13] Rośkowicz M., *Lepkosprężystość tworzyw adhezyjnych*, Mechanika Wydawnictwo Politechniki Krakowskiej, no. 3, 2009.
- [14] Rudawska A., *Wpływ sposobu przygotowania powierzchni na wytrzymałość połączeń klejowych blach ze stali odpornej na korozję*, Technologia i Automatykacja Montażu, no. 3, pp. 36-40, 2010.
- [15] Kłonica M. and Kuczmaszewski J., *Badania porównawcze wytrzymałości na ścinanie zakładkowych połączeń klejowych po oczyszczeniu mechanicznym i ozonowaniu*, Technologia i Automatykacja Montażu, no. 4, pp. 45-48, 2011.
- [16] Di Scalea F.L., Matt H., Bartoli I., Coccia S., Park G., Farrar C., *Health Monitoring of UAV Wing Skin-to-spar Joints using Guided Waves and Macro Fiber Composite Transducers*, Journal of Intelligent Material Systems and Structures, Vol. 16, pp. 373-388, 2007.
- [17] Sulejmani S., Sonnenfeld C., Geernaert T., Luyckx G., Mergo P., Urbanczyk W., Chah K., Thienpont H., Berghmans F., *Disbond monitoring in adhesive joints using shear stress optical fiber sensors*, Smart Materials and Structures 23, 2014.
- [18] Dao P.B., *Cointegration Method For Temperature Effect Removal In Damage Detection Based On Lamb Waves*, Diagnostyka 14, pp. 61-67, 2013.
- [19] Dao P.B., Staszewski W.J., *Cointegration approach for temperature effect compensation in Lamb-wave-based damage detection*, Smart Materials and Structures 22, 2013.
- [20] Cverna F., *ASM Ready Reference: Thermal properties of metals*, ASM International, 2002.
- [21] Ran Z., Yan Y., Li J., Qi Z. and Yang L.,

Determination of thermal expansion coefficients for unidirectional fiber reinforced composites, Chinese Journal of Aeronautics 27, pp. 1180-1187, 2014.

- [22] Korta J., Młyniec A. and Uhl T., *Experimental and numerical study on the effect of humidity-temperature cycling on structural multi-material adhesive joints*, Composites Part B: Engineering, Accepted for print: DOI: 10.1016/j.compositesb.2015.05.020.



Paweł Zdziebko MsC. Eng. is doctoral candidate at the AGH University of Science and Technology at the Department of Robotics and Mechatronics. His main research is focused at adhesive bonding technology and numerical simulations.



Jakub Korta PhD Eng. AGH University of Science and Technology at the Department of Robotics and Mechatronics. His research interests encompass multi-material structural design, optimization and numerical modelling.



Tadeusz Uhl Prof. Eng. is the head of the Department of Robotics and Mechatronics, AGH University of Science and Technology. His main research areas cover SHM, modal analysis, active vibration reduction, control systems and mechatronics.