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THE CHARACTERIZATION OF ZINC PLATED SURFACES AFTER AGED ADHESİVES

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1.Introduction

Adhesive bonding plays a significant role in contemporary material bonding technology, combing problems that belong to various fields of science, such as chemistry, mechanics, thermodynamics and physics. The process is widely applied for both unit and bulk production in different industrial branches, mainly in aviation, construction, automotive and electronics [1]. Pereira et al. [2] expressed that increasing restrictions are being imposed in terms of performance, pollution, safety and energy consumptions for the production of components and equipments, namely those of transportation vehicles. So there must be an effort to use new materials and processes to promote weight reduction. The use of adhesive bonding rather than mechanical fasteners offers the potential for reduced weight and cost [3].

Heller et al. [4] adhesive bonds have emerged as a particularly promising technology because of their low weight, low cost and ease of assembly. The increased usage of adhesive joints has created the requirement for more reliable techniques capable of non-destructively characterizing these types of bonds. They used methodology by guided Lamb (plate) waves to characterize adhesive bond properties.

Application of adhesive joints is usually independent to metallic substrate material (adherend). Strength of metallic adherends adhesive joint is main function of adherend surface treatment. High strength adhesive joint usually need not expensive technology, there is necessary good surface pre-treatment. Surface pre-treatment consist from several steps that prepare metallic surface to connection to polymeric glue. Typical surface pre-treatment steps of metals are [5]:

- degrease
- modification of surface roughness
- increasing of corrosion resistivity of surface
- production of conversion layer
- adhesion promoter or primer application.

Some metals and/or technology of o adhesive jointing enable consolidate or skip some surface treatment steps. Typical surface pre-treatments of metallic surface for adhesive joints are recommend in standard ČSN EN 13887 [5].

Adhesives must be resistant to degradation by environment. Adhesive layer must be impermeable for corrosion environment or, for (water) permeable, surface treatment of metal must provide sufficiently corrosion resistant layer (coating). The adhesives should be tested for corrosion resistivity and environment stability because both temperature and humidity play significant role in the strength and durability of adhesives bonds. For these reasons, in this study, we aimed to make characterization of zinc plated surface after aged adhesives. In accordance with, this analysis were carried out by using analytical scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX).

2.Experimental

- Materials

Three commercial types of adhesives were selected for this study. Sikaflex®-552 is an one-component polyurethane hybrid adhesive supplied by Sika (Sikaflex® hybrid technology combines the high performance of Sikaflex polyurethane systems with silanes, which gives great adhesion with little or no surface preparation, removing the necessity to use a primer) [6]. The second adhesive is Sikasil®SG-20, it is one component silicone based adhesive. The adhesive is moisture-curing and UV resistant. One of the main applications of this adhesive is structural glazing. According to the product data sheet, the tensile strength is approximately 2.2 MPa and the elongation at break 450% [7]. The other adhesive was chosen as Polyurethane glue Sikaflex®-263. Flexible adhesives, such as Sikaflex-552 and Polyurethane have low glass transition temperatures and low elastic modulus, but high extensions to failure [6].

In this study, zinc plated-steel (S255) were investigated in the experiments. The chemical composition of S255 is given in Table 1 based on technical sheets [8]. Surface pretreatment of zinc plated steel consist of degreasing by ethanol and application of SIKA Activator-205.

Table 1 Chemical composition of S255

Composition [wt.%]								
C	Mn	Si	P	S	Cr	Ni	N	Cu
max. 0.22	max. 0.65	0.15-0.3	max. 0.04	max. 0.05	max. 0.3	max. 0.3	max. 0.012	max. 0.3

- Ageing conditions

The preparation of adhesives for ageing test was carried out two steps. Firstly, these adhesives were poured on zinc plated-steel sheets (**see in Fig. 1)**. Secondly, the sheet specimens were immersed using the water bath equipment (ED Heating Immersion Circulators, Julabo) at constant temperature at 50 °C immersion in water for 0,1, 2, and 8 weeks. As seen that in the Fig. 1, the adhesives were exposed without water and exposed by water for 8 weeks later

- Characterization analysis

In this analysis was carried out for both zinc plated surface characterization and characterization of aged adhesives by using analytical scanning electron microscopy (SEM). In order to determine chemical composition of zinc plated surfaces after aged adhesives it was conducted energy dispersive X-ray spectroscopy (EDX) analysis using Tescan Vega III SB electron microscope. Also, the characterization of aged adhesives was conducted using Analytical Scanning Electron Microscope Tescan Vega III.

- Peel testing

The Peel testing is one way to characterize adhesive bonds. In this regard, the peel testing was carried out for three adhesives by using zinc plated surface in this study. Firstly, a thick layer was occurred on zinc plated surface for each adhesive (**see in Fig.1)**. In the second stage, after curing adhesives, they were peeled from surfaces by needle nose pliers and utility knife - coil around the pliers nose, perpendicular cuts through glue bead to adherent each 5 mm (for cohesion damage).

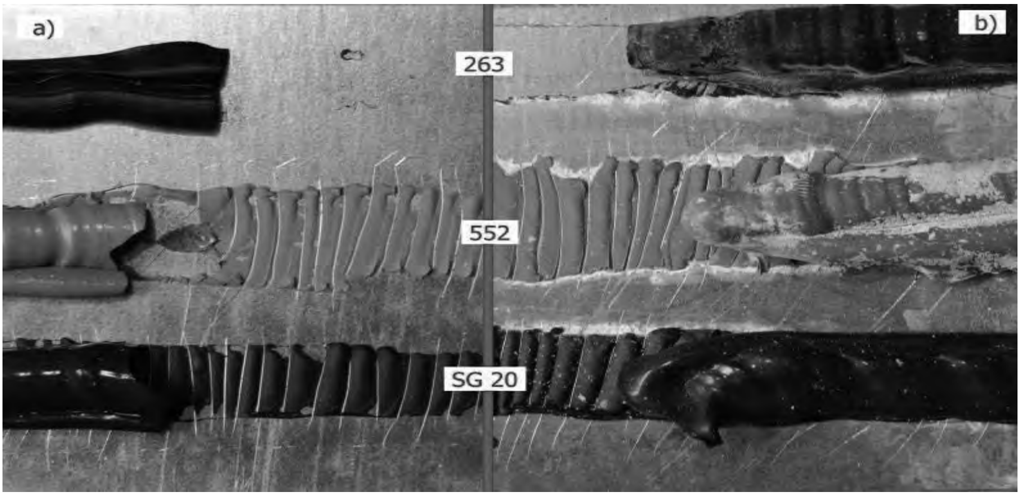

Fig. 1 *Without exposed water (a), exposed water after 8 weeks (b)*

3.Results and Discussion

• Peel testing

The selected peel test characterizes adhesion glue to adherent. A result of this test is 3 levels of adhesion – good/ partial/ none. Results of our test shows, that the adhesion of Sikaflex®-263 glue was none for our samples. The poor adhesion was observed at all stages of test.

Adhesion of Sikaflex®-552 glue was partial. After 1 week curing at normal conditions the failure was adhesive/cohesive (see. Fig. 1a). After ageing the results of adhesion tests was good – the failure of glue joints was cohesive with the exception of region of edge glue bead. On the edges of glue bead region was observed mixed adhesion/cohesion failure.

The best results of adhesion peel test show the Sikasil®SG-20 glue. Results of all tests (1 week cured, aged up to 8 weeks) was full cohesive.

- Scanning electron microscopy

SEM images were obtained at high magnification on zinc plated surfaces and under adhesive layer surface. SEM images are presented in Figs. 2 to 5. As indicated by SEM image in Fig 2, SEM indicated that the basic surface properties of zinc plated steel. Surface of initial condition of zinc plated steel do not show any corrosion – surface consist of slightly oxidised zinc. On the zinc layer was visible microcracks. This structure corresponds to standard surface of hot dip galvanising steel.

The Fig.3, Fig.4 and Fig.5 show the corrosion of surface under glues layers of Sikaflex®-263, Sikaflex®-552 and Sikasil®SG-20 adhesives, respectively. It was seen that the low corrosion resistant and the lots of corrosion interlayer products for Sikaflex®-263 in the Fig.3. As seen that in the SEM image of by using Sikaflex®-552 adhesive were observed the extremely low corrosion and visible corrosion products with small corrosion particles (Fig.4.). The last adhesive, Sikasil®SG-20 was not seen corrosion under the adhesive layer. However, the corrosion was visible on the between uncoated surface and coated surfaces in the Figure 5. The cause of failure of adhesive bonds was the presence at the interface of a layer which was itself mechanically weak. This was sufficiently thin that it could not usually be detected by means which were then available, but it was almost inevitably present [9]. According to peeling test results, Sikaflex®-263 was worst for adhesion features because the weak boundary layer was observed under the adhesive layer. Nevertheless, Sikasil®SG-20 showed best adhesion compared to others.

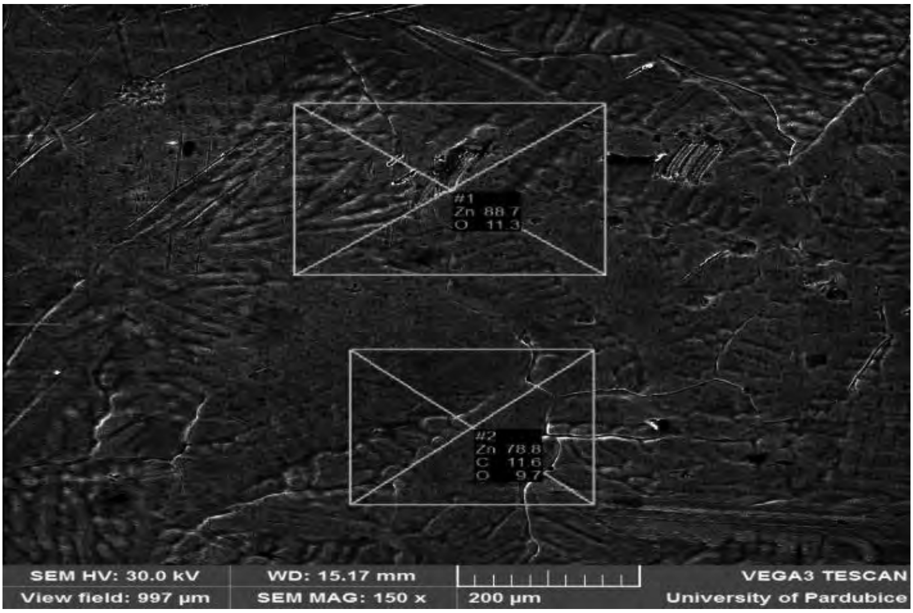

Fig. 2 *SEM image for Zinc plated surface*

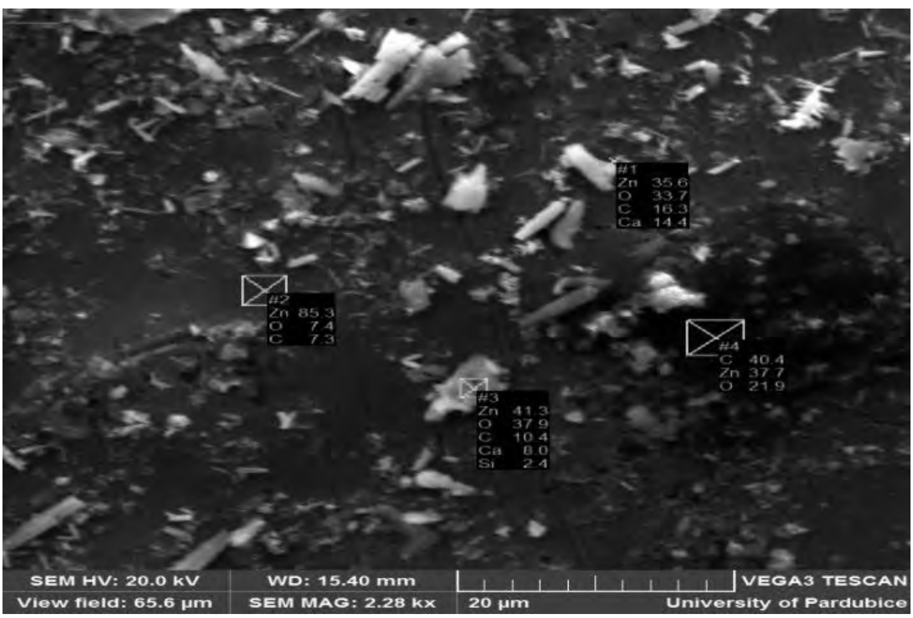

Fig. 3 *SEM image for Sikaflex®-263*

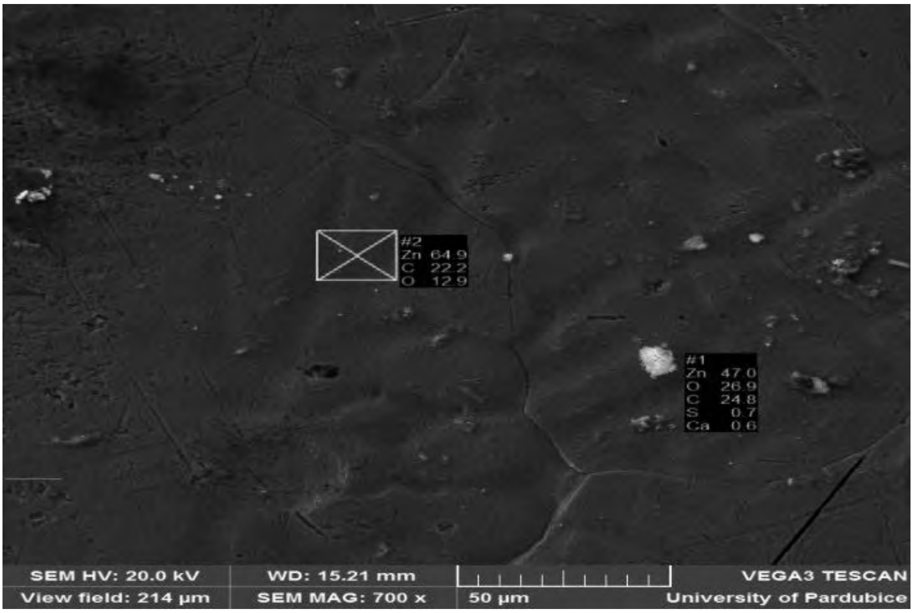

Fig. 4 *SEM image for Sikaflex®-552*

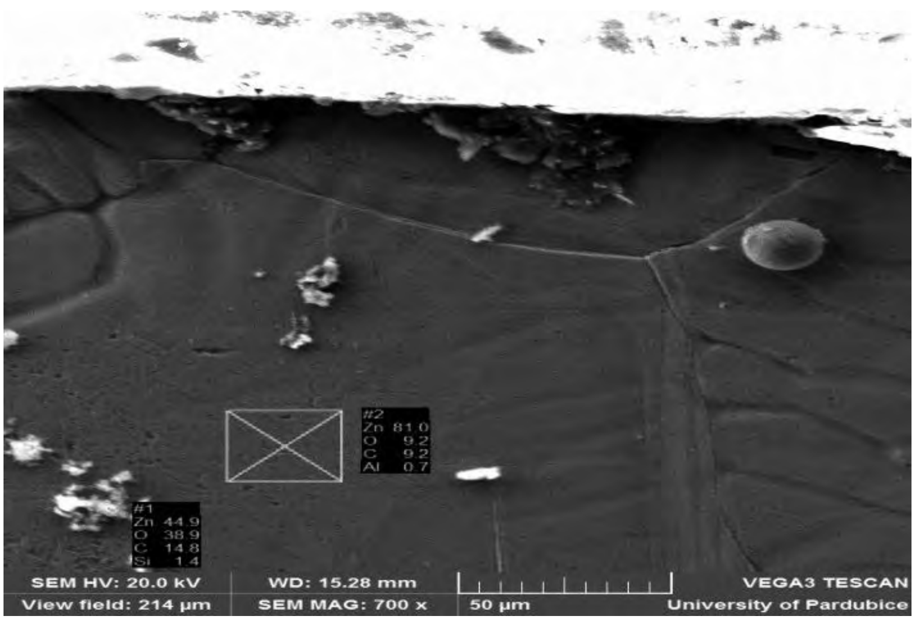

Fig. 5 *SEM image for Sikasil®SG-20.*

4.Conclusions

In this study, we have shown that characterization of zinc plated surface after aged adhesives were carried out by using peel test adhesion and SEM with EDX. A several analysis techniques will be used in the future work to characterise the surface analysis including X-ray photoelectron spectroscopy (XPS). Fo

From our results we may accept result, that the Sikasil®SG-20 adhesive would be good choosing compared to others. In case of this type of adhesion should improve the feature of corrosion resistant on zinc plated steel surfaces. Adhesion to zinc plated steel is very good even after ageing test.

Opposite of this result is the adhesion of Sikaflex®-263 glue – their adhesion was bad in all cases (cured and aged). The corrosion resistance of Sikaflex®-263 was bad, because we were found the corrosion product of zinc under glue layer after ageing of samples at water.

The Sikaflex®-552 glue shows the acceptable properties. The glue had sufficient adhesion to zinc plated steel before and also after corrosion test. The corrosion of substrate under glue layer was low. The adhesion to substrate under glue bead was good with the exception of edges of glue bead – in this area the glue show adhesion/cohesion failure after ageing test.

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Literature

1. Rudawska, A. *Selected aspects of the effect of mechanical treatment on surface roughness and adhesive joint strength of steel sheets.* International Journal of Adhesion and Adhesives, p.235-243, Vol.50, (2014).
2. Pereira, A.M., Ferreira, J.M., Antunes, F.V.,*Study on the fatigue strength of AA 6082-T6 adhesive lap joints.* International Journal of Adhesion and Adhesives, p.633-638, Vol.29, (2014).
3. da Silva, L.F.M., Ferreira, N.M.A.J., Richter-Trummer, V., Marques, E.A.S. *Effect of grooves on the strength of adhesively bonded joints.* International Journal of Adhesion and Adhesives, p.735-743, Vol.30, (2014).
4. Heller, K., Jabobs, L.J., Qu, J. *Characterization of adhesive bond properties using Lamb waves.* NDT&E International, p.555-563, Vol.33 (2000)
5. Švanda, P. *Surface Treatment of Carbon Steel for Glue Joints,* Scientific Papers of *University of* Pardubice Series B, p.195-203, Vol.18, (2012).
6. Banea, M.D., da Silva, L.F.M. *The effect of temperature on the mechanical properties of adhesives for the automotive industry.* Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials Design and Applications, p.51-62.Vol. 224, (2010)
7. Blyberg, L., Serrano, E., Enquist, B., Sterley, M. *Adhesive joints for structural timber/glass applications: Experimental testing and evaluation methods.* International Journal of Adhesion and Adhesives, p.76-87, vol. 35, (2012).
8. http://www.splav-kharkov.com/en/e_mat_start.php?name_id=884 (accessed on: 08.04.2015)
9. Allen, K.W. *At forty cometh understanding*. International Journal of Adhesion and Adhesives, p.87-93, Vol. 23 , (2003)

Summary

The characterization of zinc plated surface after aged adhesives

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The use of adhesive bonding rather than mechanical fasteners offers the potential for reduced weight and cost.This paper aims to make characterization of zinc plated surface and aged adhesives (in water at 50 °C. Surface analysis related to zinc plated surface and adhesives were carried out by using analytical scanning electron microscopy (SEM) with energy dispersive X-ray (EDX). Acoording to SEM results related to corrosion resistance which are adhesive types, Polyurethane was good, Sikasil SG20 was medium level compared to others, Sikaflex®-552 was very low corrosion resistance. As seen that in the peeling test, Polyurethane was the worst whereas SikasilSG-20 was the best. However, Sikasil® SG20 adhesive would be good choose compared to others, in case of this type of adhesion should improve the feature of corrosion resistant on zinc plated steel surfaces.