MANAGER'S SUMMARY

ZCO-61, "Improvement of Galvanneal Coating Adherence on Advanced High Strength Steel"
Progress Report No. 1
June 2008-July 2009, Issued: September 2009

The subject report describes the first year's work in this project. A comprehensive literature review was carried out and is shown in Appendix 1. It was noted that most of the work to improve coating adherence of galvanneal has been done on interstitial free (IF) steels, with little investigation of multi phase advanced high strength steels (AHSS). The materials and processing variables expected to be of highest interest for investigation were found to be: the surface roughness, the nature and amount of gamma/gamma-1 phases at the substrate/coating interface and the makeup of phases existing over the gamma/gamma-1 layer. The latter naturally correlates with the coating iron content that has been historically correlated with galvanneal adhesion performance. The chemical composition of the steel substrate is also a key variable and differences between the IF and more complex AHSS grades are of interest to the study.

Two initial series of galvanizing simulator trials are reported. The first used the University of Aachen Iwatani Rhesca simulator with an infra-red galvannealing furnace. Although samples coated well, variations in emissivity resulted in variations into the degree of galvannealing over the sample surfaces. This was also related to variations in coating thickness related to sample bending and sample positioning in the simulator. Further trials to optimize the combination of recrystallization annealing and the galvannealing treatment will be conducted subsequent to this report.

Preliminary trials were held with an induction (Trebel) simulator utilizing a helical induction coil. Samples used with the simulator had fully solidified coatings prior to galvannealing with encouraging results. Additional samples will be carried out subsequent to this report.

Final supply of both IF and AHSS steels for galvanizing simulator work has yet to be settled but it is expected to be fully resolved in the next reporting period.

Significant work was also carried out on adhesive bonding behavior of galvannealed samples using both lap shear and peel tests. Five epoxies were chosen for testing with steels donated by TKS and US Steel. In some of the shear tests, a backing strip was used to stiffen the steel sheets so that the yield stress of the steel does not influence the test results. All of the adhesives tested failed cohesively in the peel test. Use of spacers was required during the shear test to produce a shear load. Without spacing, peel stresses developed, resulting in premature cohesive failure of the adhesive or partially delaminating the GA coating. The modified shear test, with backing plates, provided discrimination between adhesive behaviors and three of the adhesives were selected for further investigation. Further consideration of adhesive test results indicated that the two adhesives, Monopox and Terokal were the best for inclusion in further adhesive testing work.

We are grateful to TKS and US Steel for their supply of samples used in the project to date.

9/09

ZCO-61: “Improvement of Galvanneal Coating Adherence on Advanced High Strength Steel”

ZCO-61-1: “Improving Joint Performance by Optimizing Galvanneal Coating Adherence”

Department of Ferrous Metallurgy (IEHK) & Welding and Joining Institute (ISF)
RWTH Aachen University

Progress Report No. 1, June 2008 - July 2009

By

Dipl.-Ing. Frédéric Huber (IEHK)
Dipl.-Ing. Sven Scheik (ISF)
Dr.-Ing. Markus Schleser (ISF)
cand. ing. Louis Souffront

Univ.-Prof. Dr.-Ing. Wolfgang Bleck (IEHK)
Univ.-Prof. Dr.-Ing. Uwe Reisgen (ISF)

Executive summary

Within this program, ZCO-61-1 “Improving Joint Performance by Optimizing Galvanneal Coating Adherence”, the purpose is to determine the effects of details of galvanneal characteristics on adhesively bonded joint shear strengths. The objectives of this project are:

- to first do a broad review of the importance of each factor known to influence galvanneal coating adherence,
- to select and process then a number of varieties of galvanneal that will be purposely made to give superior bond strength using advances high strength steels (AHSS).

This first progress report exposes primarily the realized review of literature on galvanneal coating adherence relative to adhesively bonded peel and shear strengths. Purpose is the review of the importance of each factor known to influence galvanneal coating adherence, especially the effect of steel substrate like substrate nature and alloying elements but also topography, morphology and roughness. Additionally, the nature, amount and make up of Fe-Zn intermetallic phases also play a relevant role on the galvanneal (GA) behaviour and properties, as well as the coatings Fe content and process parameters. Experimental investigations are suggested based on the literature review.

Then, in order to produce galvannealed samples for adhesive bonding, preliminary continuous GA trials using hot dip galvanizing simulator at IEHK are presented. Complete galvanizing simulation cycles, during which galvannealing occurs without intermediate solidification of the zinc coating, were carried out to investigate galvanneal coating quality with the infra-red (IR) heating furnace. Also, preliminary GA trials with intermediate Zn solidification using an induction furnace were carried out and are presented in the report. Thereafter, the selection of steel grades for the galvannealing investigation is discussed.

Concerning adhesive bonding and selection of adhesives, the characterization of applicable adhesives are presented. Mechanical characterizations were carried out using simple and modified shear tests of bonded joints, fracture pattern analysis and peel test. Moreover, influence of the adhesives curing temperature was studied. The adhesives were also characterized through thermal characterization using Differential Scanning Calorimetry (DSC) analysis and rheological characterization. These investigations result in the selection of 2 adhesives showing the highest potential for this project.

Finally, conclusions and the work program for next period are listed.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Introduction

Although hot dip galvanneal coatings are known to provide excellent corrosion performance in painted condition for automotive applications, the adherence of the coating, by the nature of its formation during the interdiffusion of iron and zinc, still poses a challenge to obtain coatings of good adherence. Improvement of galvanneal coating adherence will significantly improve the performance when using galvanneal coatings.
Based on several prior ILZRO projects about the nature of galvanneal adhesion and also on the advanced high strength steel (AHSS) grades selected for auto body and structure concept design, ILZRO set up a research programme, ZCO-61 “Improvement of Galvanneal Coating Adherence on Advanced High Strength Steel” to examine critical issues that will enhance the coating adherence of galvannealed AHSS grades. Further performance improvements of adhesive bonded joints utilizing galvannealed coatings, together with improved performance during forming operations, are expected.

In a first step within this program, ZCO-61-1 “Improving Joint Performance by Optimizing Galvanneal Coating Adherence”, the basic objective is to determine the effects of details of galvanneal characteristics on adhesively bonded joint shear strengths.
Although much work has been done on improving the quality of galvanneal, especially powdering weight loss during forming operations, much less work has been done on improving adherence strength in joints where high shear and peel strengths are required.
The purpose of this project is to first do a broad review of the importance of each factor known to influence galvanneal coating adherence and then to select a number of varieties of galvanneal that will be purposely made to give superior bond strength based on this review. Samples will be prepared in a galvanizing simulator to allow testing of samples for shear and peel strength in the undeformed condition.

Through this work, RWTH Aachen University wants to investigate the influence of the sheets surface on the zinc layer properties in combination with optimal adhesive bonding in order to match adhesive bonding with coating systems. A reliable and reproducible adherence layer bond by different strip surface topographies should be reached.
According to the ZCO-61 research plan, following tasks will be carried out within the work program:

- Task 1: Review of literature on galvanneal coating adherence relative to adhesively bonded peel and shear strengths
- Task 2: Production of galvannealed samples for adhesive bonding
- Task 3: Shear strength testing of adhesively bonded samples
- Task 4: Microstructural and chemical analysis of high quality samples
- Task 5: Analysis and reporting

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Task 1: Review of literature on galvanneal coating adherence relative to adhesively bonded peel and shear strengths

A literature survey was realized in the framework of this project. The entire literature review (70 pages) is presented in **appendix 1**.
The summary and prospects is presented below.

The adherence of the galvanneal coating to the steel sheet is influenced by several factors such as:

- steel chemistry,
- crystallographic features of the growing galvanneal coating,
- presence of cratering,
- elements composition in the galvanneal layer,
- depth of preoxidation, grain sizes and textures in the steel substrate.

All these factors are influenced by steel pre-treatment variables, the aluminium content of the galvanizing bath, the steel chemistry and the galvannealing heat cycle parameters. Many research papers describing the above separate effects were published. Within several ILZRO projects, the nature of galvanneal adhesion has been investigated, including projects ZCO-12, ZCO-14 and ZCO-51.
Purpose of this task is first to do a broad literature review of the importance of each of the mentioned factors and any others known to influence galvanneal coating adherence, especially the effect of steel chemistry and galvanizing/galvannealing annealing parameters. Adherence is measured by both peel and shear strengths of bonded joints and also by measures of powdering and flaking.
Then, based on the literature review, a number of galvanneal coating structures and chemistries that will be likely to give the best shear and peel strengths in adhesively bonded joints should be selected, together with steel chemistries, textures and processing routes likely to produce these galvanneal microstructures.

Knowing the importance of each factor known to influence the GA coating adherence relative to shear strength of bonded joints would be very helpful to make a selection of a number of GA coating structures and chemistries for experiments that will be likely to give the best shear strength.
This broad literature study underlined some factors related to the steel substrate influence like substrate nature and alloying elements but also topography, morphology and roughness. Additionally, the nature, amount and make up of Fe-Zn intermetallic phases also play a relevant role on the GA behaviour and properties, as well as the coating Fe content and process parameters.

The **chemistry of steel substrates** strongly affects the initial reactivity and the formation and development of intermetallic phases and their distribution in galvannealed coatings, even at the same average Fe coating content.
For instance it has been reported that C, Si, and P in steel restrain the alloying reaction between Fe and Zn, whereas Ti and Nb promote the reaction between Fe and Zn.
The presence of excess Ti in the IF steel substrate reduces the bonding strength. Concerning Ti-Nb IF steels, they were shown to yield excellent galvanneal coating adhesion properties

Univ. Prof. Dr.-Ing. Wolfgang Bleck
www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Fon +49 241 80 95783
Fax +49 241 80 92224

and higher shear strength values (better than Ti IF). Small Si-additions have been reported to be very effective in improving the stone chipping resistance and the adhesion strength of galvannealed steel. Si reduces the growth of the Γ layer.
Phosphorus is beneficial for producing a galvanneal coating with higher interfacial shear strength: P favours the nucleation of the Γ_1 phase and effectively restrains the nucleation of the Γ phase.
From the investigation of galvannealing behaviour, it was found that the galvannealing rate of developed AHSS is equal to that of mild steel. The increase in Si sharply decreases Fe% in the coating and also has significant negative effects on the coating uniformity.
Galvannealed coatings on high strength IF and non-IF steels are characterised by higher adhesion strengths, the average adhesion strength on the high strength steels being always higher (up to 8MPa).
However, most literature is dealing with IF steel substrates and only few citations deal with AHSS. The IF steel having the highest potential for lap shear test of adhesively bonded GA steels is a Si-added Ti-Nb IF steel grade. Concerning multiphase AHSS like DP or TRIP, no prediction can be issued so far. Thus, ILZRO sponsors should be asked through a survey about the AHSS grades they'd to supply for experimental investigations. These highly alloyed substrates will help to investigate the influence of alloying elements combinations on the coating development and make up and its behaviour during lap shear test.

The dependency of **Fe-Zn intermetallics** growth behavior on the crystal orientation of the base IF steel was clearly observed. In general, ζ crystals are precipitated orderly on (111) α, whereas ζ crystals are precipitated disorderly on (001) α and (101) α at the outset of the formation of Fe-Zn intermetallics, and the growth rate of Fe-Zn intermetallics on (001) α and (101) α is larger than that on (111) α. This phenomenon has an influence on the subsequent development of the other Fe-Zn intermetallic phases.
Cratering occurs preferentially at low galvannealing temperatures as well as low "galvannealing-reactivity" steel substrates. The dimensions of craters are governed by the substrate grain size. The enhanced Fe consumption along the grain boundaries and the lower consumption of iron on {111} α-Fe grains increase the steel / coating interface roughness, which improves its shear strength. In fact, it was found that the larger the roughness of the steel substrate, the greater the interfacial shear strength is, but also that the greater the surface roughness of the steel substrate, the thicker the coating is.
Craters don't have a negative influence on GA product user's properties, including corrosion. On the contrary, the presence of craters has a positive effect on coating adhesion properties.
Thus, investigations on the influence of the substrate roughness will be carried out in the project framework.

The formation of **Γ-phase**, which occurs preferentially at the grain boundaries of the base steel, increases the adhesion strength. The influence of Γ-phase growth and thickness is inconsistent in the literature, some references consider that increasing Γ-phase amount does not lower coating adhesion, others consider the opposite.
The formation of Γ_1-phase is suppressed in coatings with Fe contents less than 10% and grows rapidly in the coating with Fe contents ranging from 10 to 11%.
The galvanneal coating with Γ_1 phase interfacing with the substrate steel forms out at the early stage of galvannealing and has the highest interfacial shear strength. The interfacial shear strength is reduced when the Γ phase nucleates between α-Fe and Γ_1 phases. The presence of a Γ_1 phase instead of a Γ phase increases the interfacial strength.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Finally, Γ/$Γ_1$ formation seems to be dominant in respect to adhesion behaviour of galvannealed steel sheet. Therefore crater formation supposedly does not play a major role, but the influence of galvannealing temperature becomes obvious.
The coating with optimum performance is proposed with the $Γ_1$ phase interfacing with substrate steel and the softer δ phase having moderate Fe concentration as the outer layer.
Hence, the experimental investigation of the influence of the nature and amount of the Γ and $Γ_1$ phases at the substrate/coating interface would be very interesting. This could be a key for improving GA coating adhesion and behaviour during lap shear test.

High **galvannealing temperature** leads to a deterioration of the coating adhesion and to an increase of the powdering amount. In addition, the increase in interfacial roughness with galvanneal soak time may explain the observed increase in interfacial strength at higher galvannealing times. Thus, investigations should focus on low GA temperatures in order to insure satisfying coating properties like good formability and low powdering.

Powdering increases with Fe content in the coating but powdering weight loss does not increase continuously with coating Fe weight, but seems to reach a maximum around 15%. It can even decrease again beyond this value.
In fact powdering is related to the iron content of the Γ and δ mixture layer. The powdering increases with the increase of iron content in the Γ and δ mixture layer, even though the iron content of the total coating was the same. Nb, B, Mn and Si appear to prevent iron accumulation in the Γ and δ mixture layer.
The increase in the percentage of $Γ_1$ phase in the coating improved the powdering resistance.

In order to insure pure shear testing and absence of plastic deformation of the substrate, a **lap shear testing** method with backing giving the shear strength of the substrate / coating interface independently of the mechanical characteristics and thickness of the substrate is needed.
Measured shear strength depends critically on the type of adhesive and the geometry of the overlap zone. Moreover, the shear strength can be influenced by the Fe content of the galvannealed layer, the effect of steel substrate alloying elements and the Fe-Zn phase distribution within the coating.

Coatings with low **coating Fe content** present high lap shear strength due to the presence of the ductile η phase. Remarkable lowering in shear strength can be seen in the coatings with approximately 7 to 11% Fe. At higher Fe contents, high lap shear strength is observed and seems to result from the lateral growth mode of the Γ-phase, which is associated with a ledge formation mechanism. Adhesion strength can also be related to the position of the rather complex fracture plane; at high Fe content, the fracture plane is almost exclusively located at the steel/ Γ phase interface.
However, the Fe composition dependence of the adhesion strength of galvannealed may not be the best way to evaluate the real cause of the improvement in adhesion strength. This is due to the fact that the Fe-content of the coating is not homogeneous.
It would be better to consider just the relative phase make up and composition of the coatings instead or additionally to the Fe content dependence of the GA adhesion and shear strengths. Also, when considering coating Fe content, investigations concerning the suitable content, i.e. low or high, should be targeted in order to reach improved strength in accordance with satisfying powdering resistance.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Please find in **appendix 2** a survey concerning GAP sponsors requests and comments (steel grades to be investigated, GA process parameters…)

The **adhesives** used in the project first of all have to develop sufficient adhesion forces to be able to test the zinc coating. Habenicht et. al. claims that the strength of adhesively bonded zinc coated steel sheets is determined by the adhesion strength of the adhesive/steel surface interface, respectively the zinchydroxid- and zinccarbonate layers. The anticorrosive layers on the galvannealed surface have a strong connection to the base material. Furthermore epoxy resin based adhesives are able to develop strong adhesion forces to the zinchydroxid- and zinccarbonate layers by chemical and intermolecular bonding.

Lin et al. claims that the roughness of the galvanneal coating favours adhesive bonding since micro cavities increase the effective surface area. Habenicht says that the micro-morphology of the oxide formation is a 3-dimensional system and increases the effective surface and consequently the reactive contacts. Thus adhesives should be considered which have a rather low viscosity at curing temperature.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Task 2: Production of galvannealed samples for adhesive bonding

Preliminary continuous GA trials

The study samples should be galvannealed using the hot dip process simulator available at the IEHK. A complete galvanizing simulation cycle, during which galvannealing occurs without intermediate solidification of the zinc coating, is expected to be proceeded. The annealing and galvannealing process variables (annealing cycle, galvannealing temperature…) will be chosen respectively to the recommendations of task 1 and experience.

The galvannealing process should be realized using the infra-red heating furnace of the simulator (heating ramps up to 50K/s). Annealing subsequent to hot dip galvanizing cannot so far be realized continuously using an induction heating furnace (heating ramps up to 150K/s) typically used for galvannealing and during industrial process.
On the one hand, it was reported that the alloying reaction for induction heating is well in advance of that for infra-red heating with absence of outbursts in the corresponding induction condition. Apart from the differences in the relative degree of alloying, no major microstructural and morphological differences could be discerned between the simulation methods (IR and induction) and various materials examined.[1]
On the other hand, it was reported that the homogeneity of the coating thickness and Fe-Zn phases is much better when induction heating is used, the appearance of the outbursts structures being independent on the heating method. Moreover, using the induction furnace, it is possible to have a real “square” T-t cycle for GA.[2]

[1] G.A. Jenkins, L.J. Burrows: Investigation of Zinc/Iron Alloying in Various Commercial Galvanised Steels during Experimental Galvannealing by Radiation and Induction, Galvatech´92, 1992, 209 – 214
[2] M.J. Volt, F.J. Alonso: Improvement in hot galvanising and galvanealing processes and product properties, European Commission, 2002

Hence preliminary trials have to be carried out to insure that a sufficient galvanneal coating quality can be reached with the infra-red (IR) heating furnace. If the characteristics of the infra-red heating furnace of the simulator are too weak for galvannealing without intermediate solidification of the zinc coating, annealing of previously galvanized samples of the correct characteristics will take place in another heating furnace of the IEHK. Otherwise, IEHK is counting on the acquisition of an induction heating system for the hot dip process simulator in the framework of another HDGA project, but IEHK has been waiting for the cooperation agreement since end of 2008.

The experiments were realized at IEHK using a hot dip process simulator (called Rhesca) HDPS EU AII from Iwatani modified by IEHK.
The steel grade DC01, Table 1, with 0,75mm thickness was used for the experiments. The sample size is 120mm x 200mm.

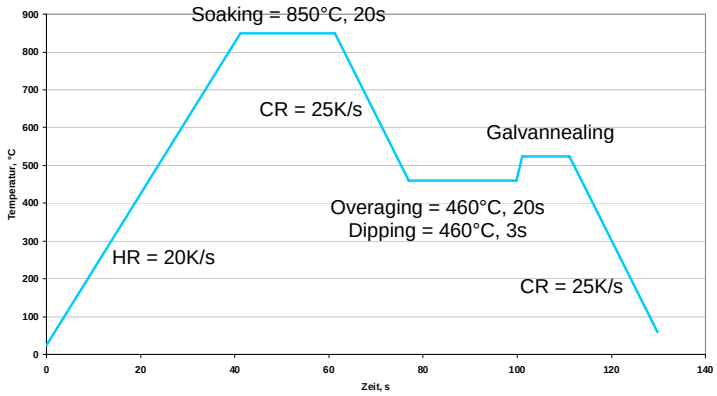
DC01	C	Si	Mn	P	Cr	Mo	Ni	Al	Nb	Ti	B
wt%	0,057	0,013	0,262	0,006	0,015	<0,005	0,012	0,033	<0,001	<0,001	<0,001

Table 1: Chemical composition of DC01 steel

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

The process parameters for the simulation are listed below:

- Gas atmosphere: 5%H2 - 95%N2, dewpoint = -30°C
- Zn bath: ~0,135% Al, T = 460°C
- Wiping gas: N2, T= 150°C
- Sample driving speed between wiping knives and IR-furnace: 1,5m/s
- T-t cycle, Fig. 1:



GA cycle	
T (°C)	t (s)
500	5
	10
	15
	20
	30
525	5
	10
	15
	20
	30
550	5
	10
	15
	20
	30

Fig. 1: T-t cycles used for preliminary continuous GA trials.

During reheating with IR-furnace after wiping, the thermocouple measures a heating rate about 20K/s due to the surface emissivity of the shiny outer zinc surface, which lowers the heat absorption of the sample in comparison to the recrystallisation annealing step.

The surface quality of the processed samples is presented in Fig. 2. The iron diffusion into the coating is favoured by increasing GA temperature and time.
The galvanneal coating was investigated under light optical microscope, Fig. 3. The average thickness of the coating is 10µm and the microstructure was revealed using Kilpatrick etching. The Fe-Zn intermetallic phases make up is GA temperature and time dependant.
The galvanneal coating of samples galvannealed at 525°C was also investigated under scanning electron microscope (SEM), Fig. 4, and analysed using EDX, Fig. 5. Different coating make ups can be identified thanks to chemical compositions of analysed Fe-Zn phases.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

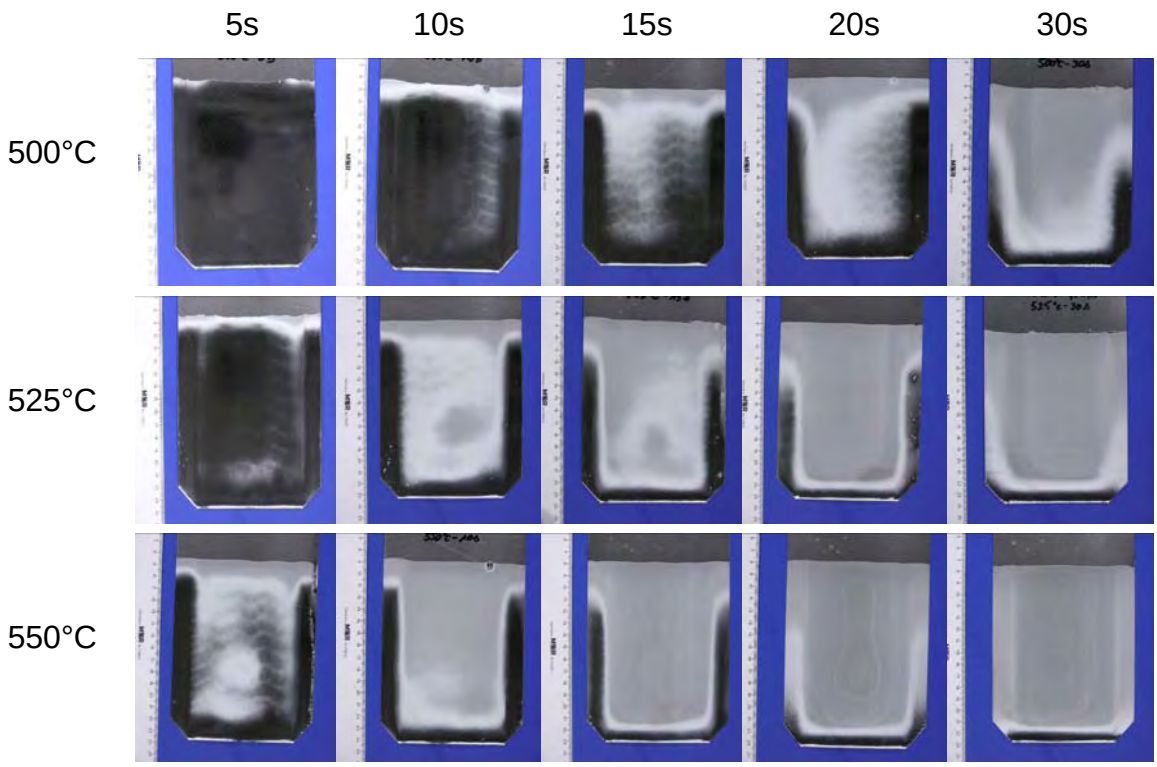


Fig. 2: Surface quality after the first preliminary continuous GA trials.

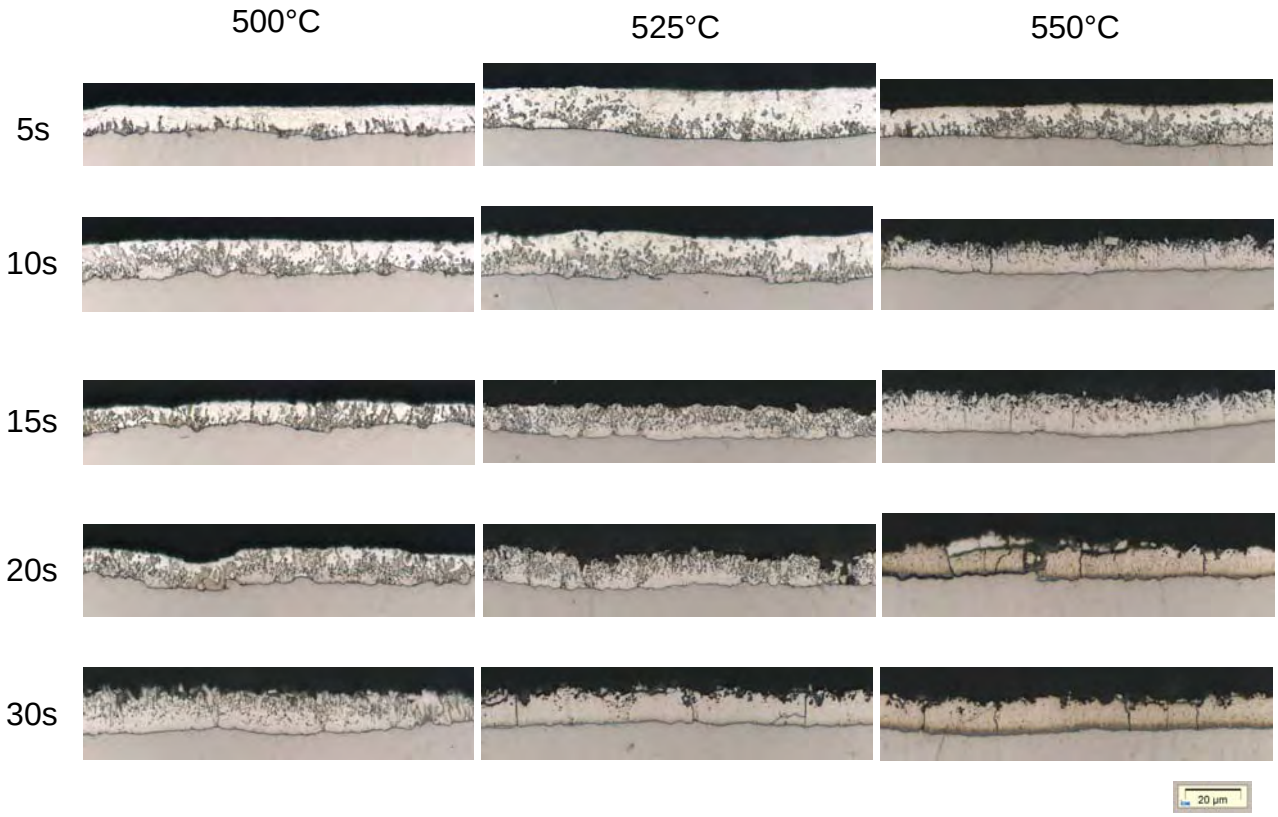


Fig. 3: Cross-sections of galvanneal coating under light optical microscope (1000x magnification, Kilpatrick etching).

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

GA with T = 525°C

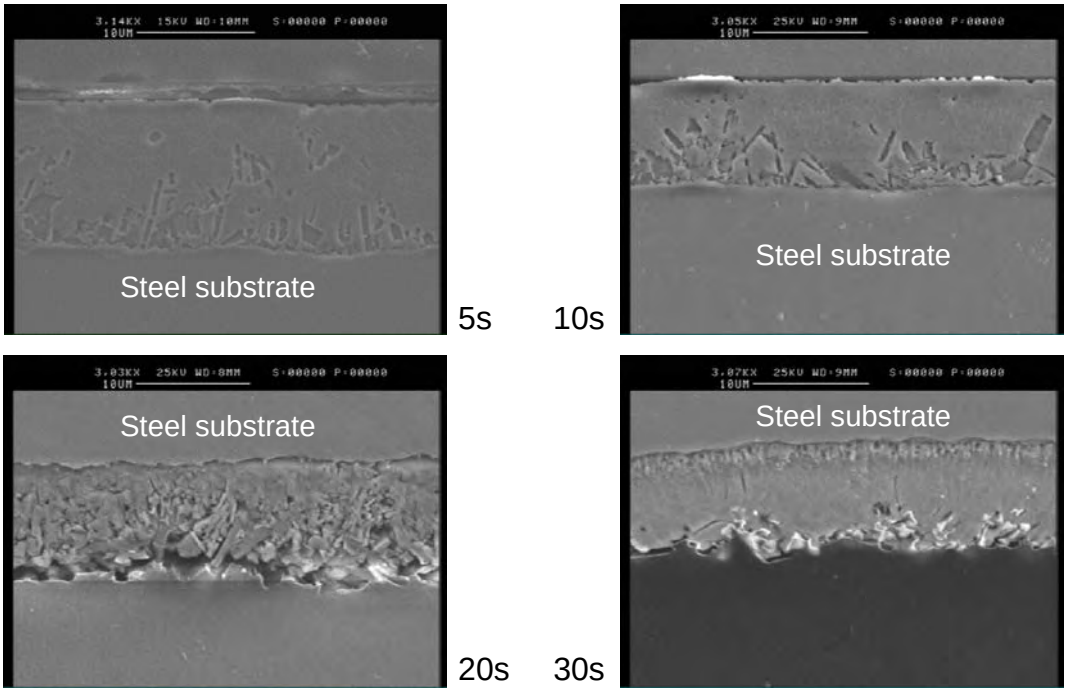
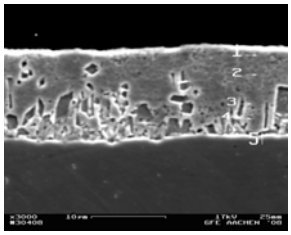


Fig. 4: Cross-sections of galvanneal coating galvannealed at 525°C under SEM.

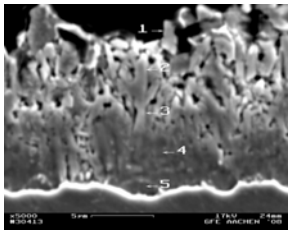
GA with T = 525°C

10s



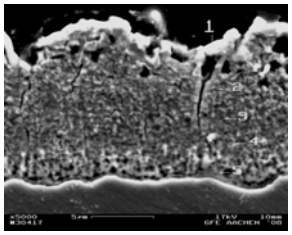
Position	Elements in wt.%			Elements in at.%			Expected phase
	Fe	Al	Zn	Fe	Al	Zn	
1	0,77	0,31	96,24	0,83	0,68	88,41	η
2	1,43	0,35	98,23	1,66	0,84	97,51	η
3	1,59	0,40	95,85	1,73	0,90	89,15	η
4	3,02	0,12	95,15	3,33	0,27	89,79	η
5	8,85	0,67	87,79	9,35	1,47	79,27	δ

20s



Position	Elements in wt.%			Elements in at.%			Expected phase
	Fe	Al	Zn	Fe	Al	Zn	
1	8,18	0,53	87,97	8,51	1,14	78,25	δ
2	7,17	0,57	90,52	7,81	1,28	84,28	δ
3	9,02	0,51	88,43	9,72	1,13	81,43	δ
4	9,50	0,42	88,48	10,38	0,94	82,57	δ
5	16,89	0,24	80,88	18,08	0,53	73,95	Γ1

30s



Position	Elements in wt.%			Elements in at.%			Expected phase
	Fe	Al	Zn	Fe	Al	Zn	
1	9,98	0,84	82,84	9,54	1,66	67,64	δ
2	9,18	0,55	87,27	9,63	1,20	78,21	δ
3	9,87	0,94	86,32	10,32	2,03	77,15	δ
4	11,15	0,56	84,91	11,54	1,19	75,05	δ
5	21,45	0,15	71,52	20,07	0,30	57,17	Γ / Γ1

Fig. 5: Cross-sections of galvanneal coating galvannealed at 525°C analysed using EDX under SEM.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

These investigations underline:

- an inhomogeneous diffusion repartition on the whole sample, mainly for low GA temperatures or short GA times, Fig. 2,
- an inhomogeneous diffusion between the both samples sides,
- presence of outbursts at the early stages of diffusion.

It can be assumed that these undesired properties can be due to surface emissivity variations of the shiny outer zinc surface or perhaps due to non-uniform GA heating or a too slow heating rate to insure a homogeneous GA diffusion.

During the GAP meeting in November 2008, it was suggested to investigate:

- Zn-coating thickness distribution before GA,
- temperature distribution induced by the 3 zones IR-furnace by using 3 thermocouples.

The coating thickness measuring device used is a Erichsen eXacto Typ F (for measurements on steel) using magnetic induction principle, Fig. 6.
2 different measurements methods were carried out:
- Points measurements: 9 points and 5 measurements per point, on each sheet side, Fig. 6,
- CW map: grid 1cm x 1cm and 10 measurements per point, on each sheet side.

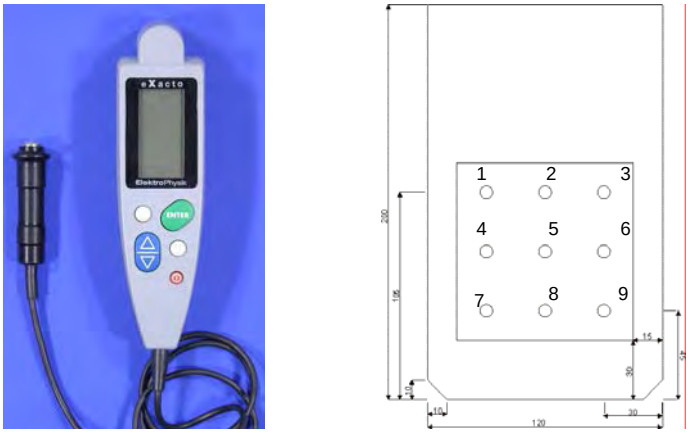


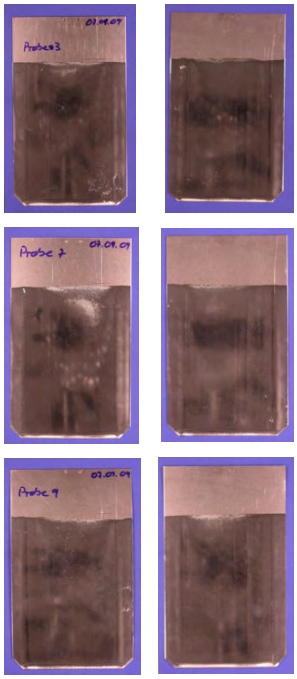
Fig. 6: Erichsen eXacto Typ F and points measurements method.

Coating thickness point measurements were performed on DC01 samples: 3 GI samples and 3 GA samples (GA parameters: 525°C, 20s). The sample side with a thermocouple is denominated as side A and the other sample side as side B. The measurements are presented in Fig. 7 and Fig. 8.

Coating thickness CW map measurements were performed on DC01-GI samples. The sample side with the thermocouple is denominated as side A and the other sample side as side B. The measurements are presented in Fig. 9.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Side A (with TC) Side B



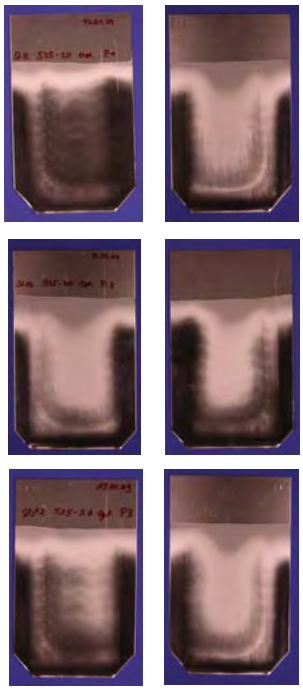
Sample 3	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	6,2	5,9	5,8	7,1	6,0	5,7	6,0	6,3	6,5
Side B	7,4	9,3	10,0	9,0	8,0	9,0	8,4	8,4	9,1

Sample 7	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	5,8	6,6	6,8	7,0	7,0	7,2	8,0	7,8	7,4
Side B	6,7	6,9	8,5	7,4	7,2	7,5	7,4	6,9	8,7

Sample 9	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	11,9	8,5	8,9	10,1	8,4	9,5	11,1	9,6	9,7
Side B	7,4	7,4	8,4	8,3	5,9	7,4	6,0	7,0	8,3

Fig. 7: Zn-coating thickness distribution of DC01-GI (before GA process).

Side A (with TC) Side B



Sample 1	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	11,5	9,6	9,5	10,8	11,1	11,2	13,4	10,3	11,2
Side B	9,0	9,3	10,7	10,1	9,9	10,7	8,9	9,1	12,0

Sample 2	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	12,4	11,6	11,3	12,9	11,7	9,5	12,2	10,1	9,3
Side B	10,0	9,2	10,5	10,7	12,8	11,5	11,4	10,8	13,2

Sample 3	1[µm]	2 [µm]	3 [µm]	4 [µm]	5 [µm]	6 [µm]	7 [µm]	8 [µm]	9 [µm]
Side A	8,7	11,9	9,0	11,6	12,0	10,9	13,9	9,8	9,7
Side B	9,7	11,8	10,9	10,1	9,3	12,1	10,4	9,2	12,2

Fig. 8: Coating thickness distribution of DC01-GA.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

The coating thickness point measurements method is very quick but not well suited, since the thickness deviation between both sample sides is not recognizable because of the small amount of measurements.
Indeed, the CW map method underlines a coating thickness deviation between both sample sides. The coating on one side is almost always quite thicker than on the other side, but the side dependence of the coating thickness is not related to the thermocouple welding side. The thermocouple welding side is fixed and always located on the left side of the wiping system. Thus, the wiping system seems not to be responsible for the coating thickness deviation between samples sides. Moreover, the coating on the edges of the samples is always thicker; this is simply due to the guiding device in the pot.
The rolling direction being parallel to sample width, there is some small bending resulting and implying that one side of the sample is closer to the wiping nozzle than the other side. Thus, the sample bending is responsible for the coating thickness difference between both sample sides.

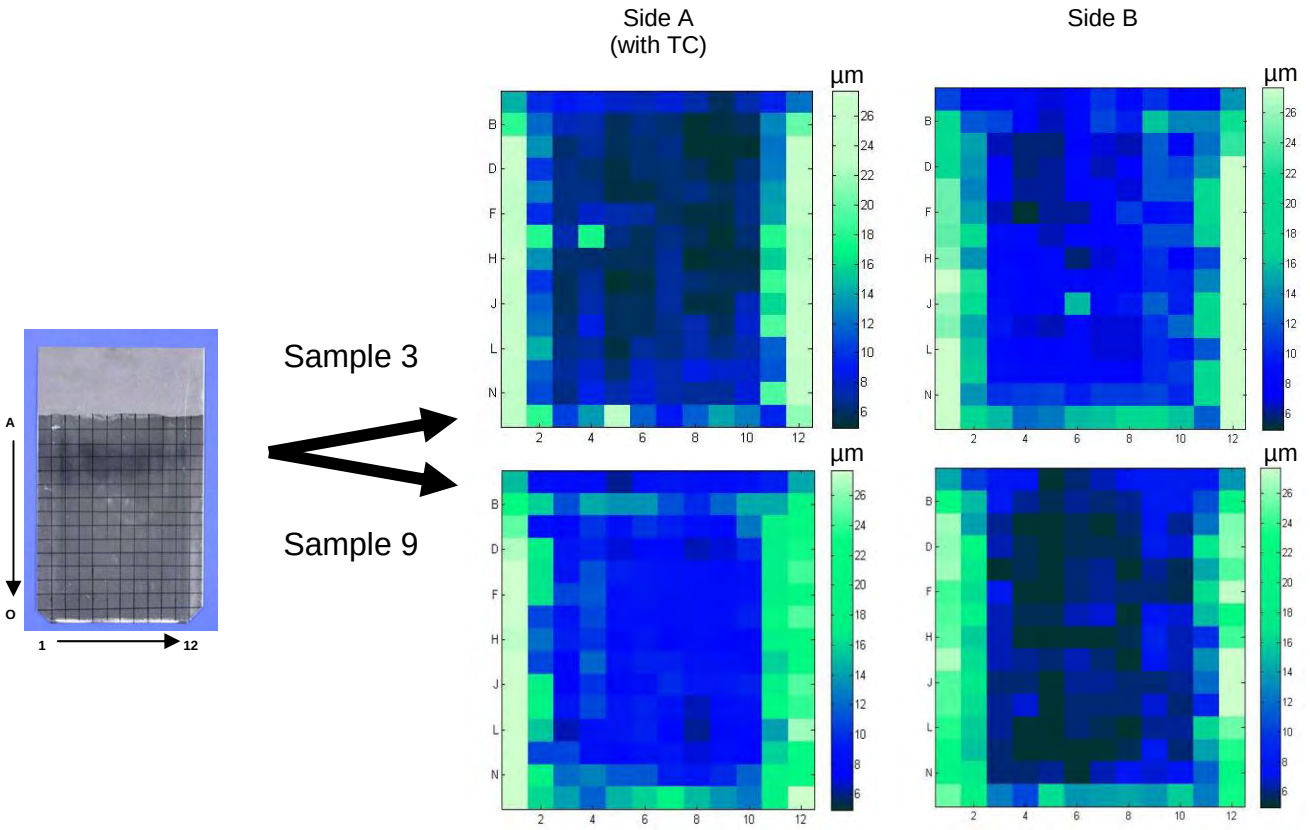


Fig. 9: Zn-coating thickness distribution CW map of DC01-GI (before GA process).

The 3 zones IR-furnace was studied with different parameters, i.e. with:

- IR-furnace zones parameters optimized for recrystallization annealing,
- identical IR-furnace zones parameters for equal radiation during GA.

IR-furnace zones parameters were optimized for recrystallization annealing using 3 thermocouples, Fig. 10. The DC01-GA samples processed with these furnace parameters (GA parameters: 525°C, 20s), Fig. 11, look like those presented Fig. 2. The DC01-GA samples processed with identical IR-furnace zones parameters for equal radiation during GA

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

(GA parameters: 525°C, 20s) are presented in Fig. 12 for 2 different furnace parameters combinations. The results are not very satisfying and inhomogeneous diffusion distribution on the whole sample and between both samples sides still exists.

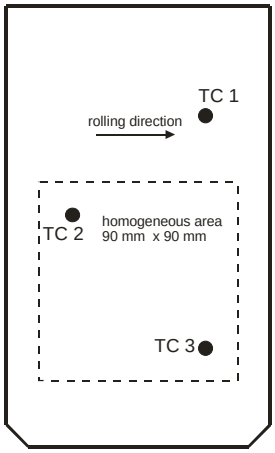


Fig. 10: Rhesca sample with 3 thermocouples.

Only one IR-furnace parameter combination is possible during an experiment with Rhesca HDPS type EU A II at IEHK. Thus, recrystallization annealing and GA-step cannot be simultaneously optimized. But, additionally trials will be carried out with varying:

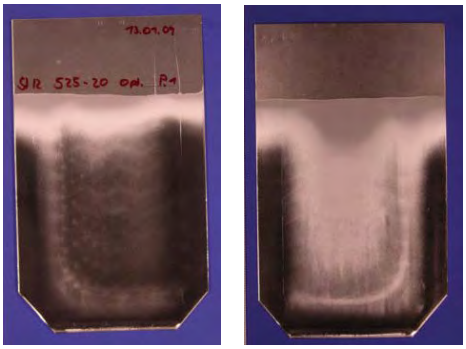
- IR-furnace parameters
- sample position in IR-heater
- sample cooling position

Hopefully a parameter combination ensuring sufficient GA coating quality will be identified, otherwise GA must be performed with intermediate solidification of molten Zn.

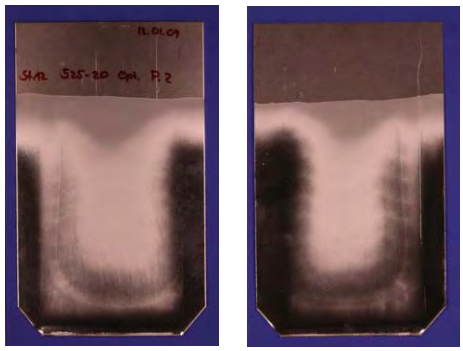
Besides, Mc Master University offers the possibility to use their HDG simulator (equipped with an induction furnace for GA) during the next months. RWTH Aachen University is very interested in this offer and will contact the person in charge.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

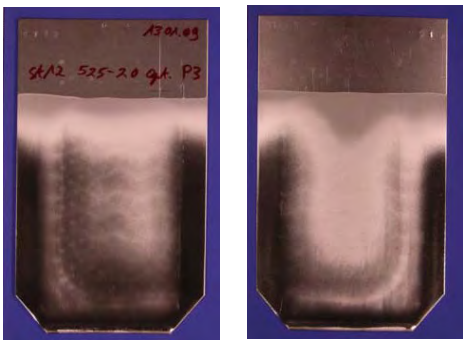
Sample 1



Sample 2



Sample 3



Sample 4

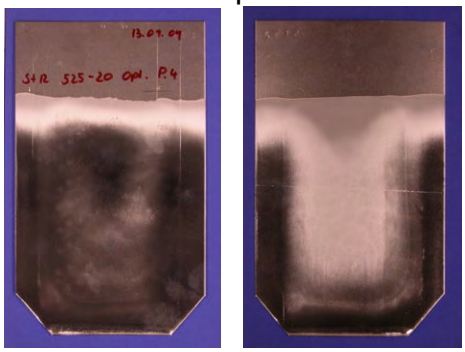
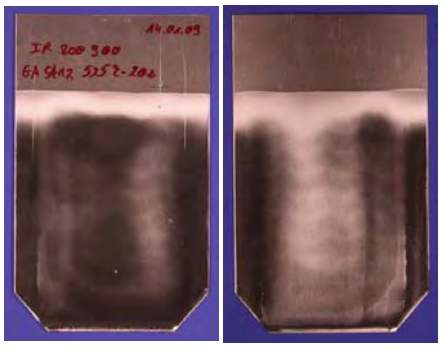


Fig. 11: GA samples processed with IR-furnace zones parameters optimized for recrystallization annealing.

IR parameters: upper zone, middle zone, bottom zone

- IR ramp: 800, 800, 800
- IR hold: 900, 900, 900



- IR ramp: 800, 800, 800
- IR hold: 1000, 1000, 1000

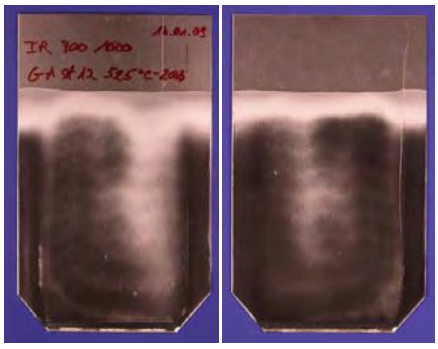


Fig. 12: GA samples processed with identical IR-furnace zones parameters for equal radiation during GA.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Preliminary GA trials with intermediate Zn solidification

Equipments for GA process with intermediate Zn-coating solidification are available at IEHK. But the Gleeble 512 simulator is an old equipment not well suited and to be put out of service (analogical output, sample side 100mm x 75mm with homogeneous annealing area 10mm x 75mm in the middle). Thus, GA trials with intermediate solidification are processed with the Trebel hot forming simulator, Fig. 13.

The Trebel simulator has a helix induction coil, temperature repartition between the edges and the middle is checked with 2 thermocouples. The homogeneous annealing area represents 25 – 30 mm in the sample middle.
Sample geometry and GA parameters are presented in Fig. 14.
Processed GA samples with intermediate Zn solidification are presented in Fig. 15.
The galvanneal coating was investigated with a light optical microscope, Fig. 16. The average thickness of the coating is 20µm and the microstructure is revealed using Kilpatrick etching. SEM/EDX comparisons between the middle and the edges are presented in Fig. 17 to Fig. 20.

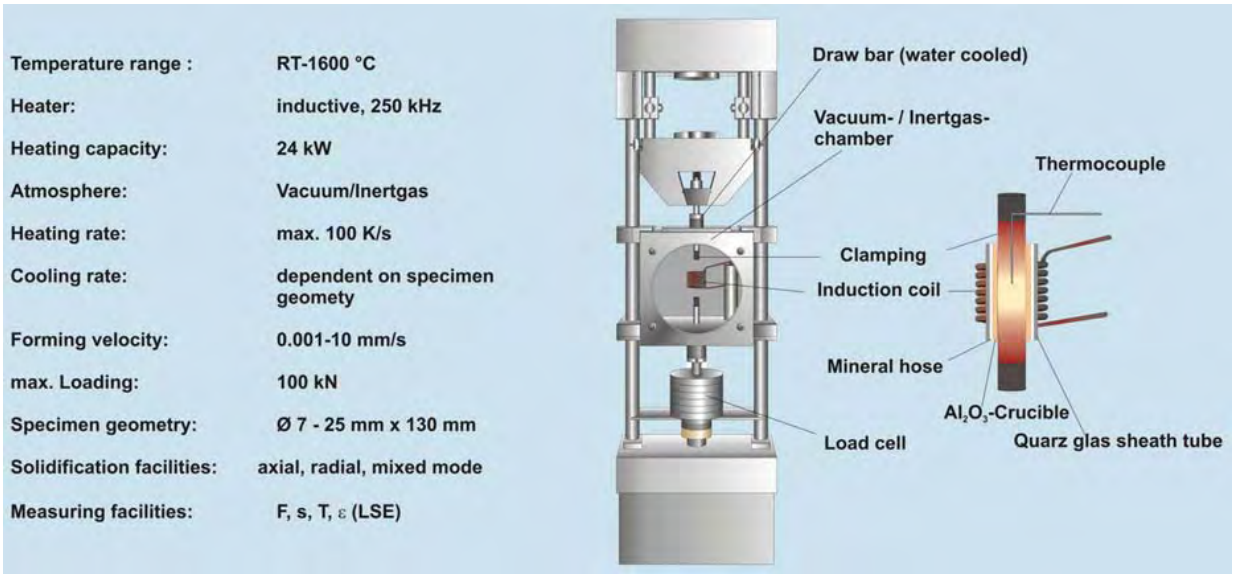


Fig. 13: Trebel hot forming simulator.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

GI sample
coated with Rhesca

150 mm

20 mm

GA process parameters

- Heating: 45-50°C/s
- Holding
  - Strip sample 1: 500°C, 20s
  - Strip sample 2: 525°C, 20s
  - Strip sample 3: 500°C, 30s
  - Strip sample 4: 525°C, 30s
- Quenching: ~130°C/s

Fig. 14: Sample geometry and GA parameters for Trebel simulations.

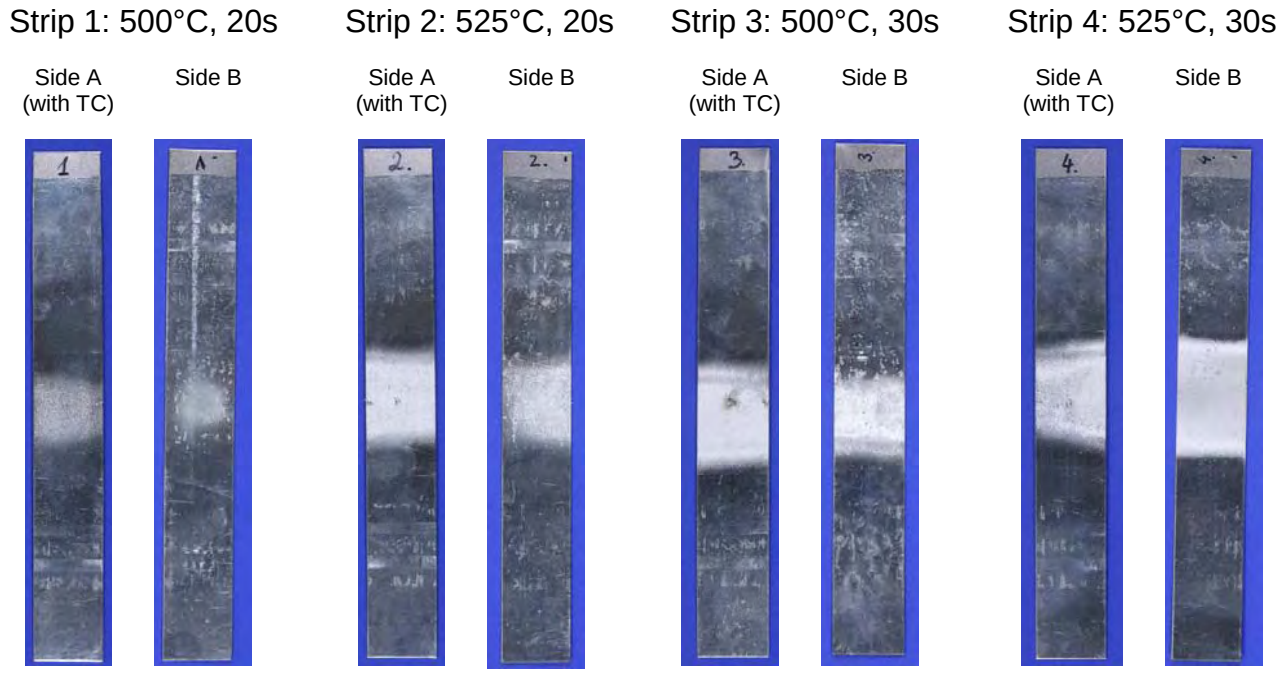


Fig. 15: Processed GA samples with intermediate Zn solidification.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

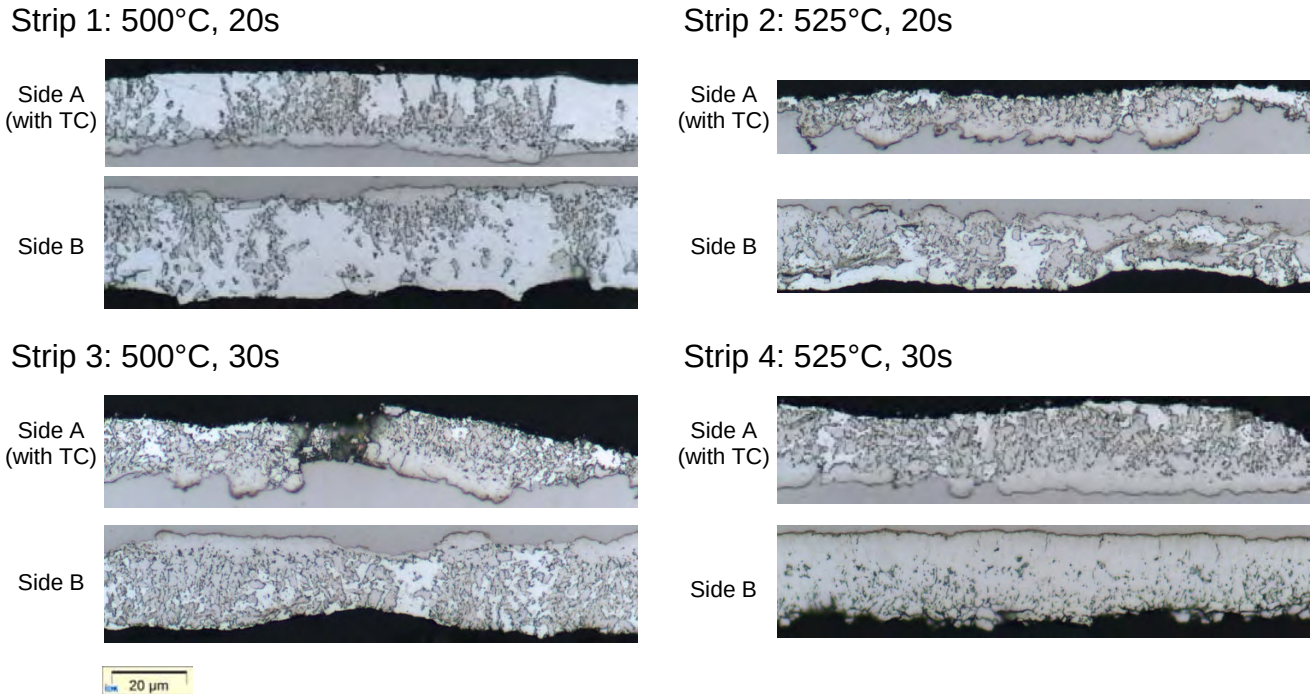
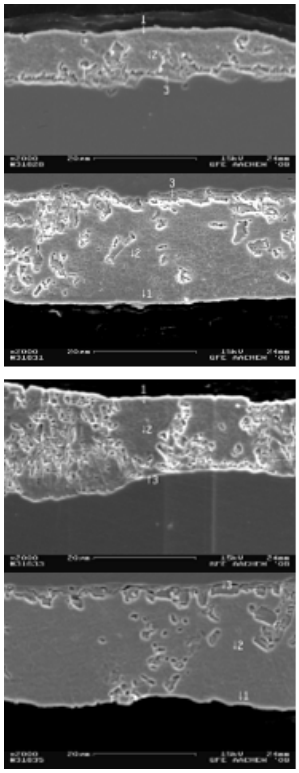


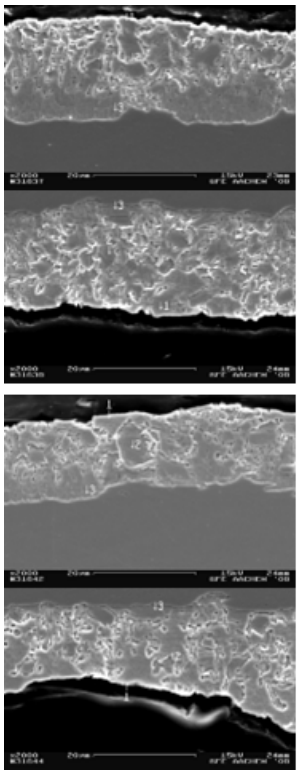
Fig. 16: Cross-sections of galvanneal coating with a light optical microscope (1000x magnification, Kilpatrick etching).



Position on sample	EDX point	Elements in wt.%			Elements in at.%			Expected phase
		Fe	Al	Zn	Fe	Al	Zn	
Center top	1	0,87	0,19	96,58	0,94	0,42	89,66	η
	2	1,45	0,26	96,74	1,61	0,59	91,76	η
	3	7,63	1,29	88,53	8,05	2,81	79,76	δ
Center bottom	1	0,27	0,27	97,61	0,30	0,61	91,96	η
	2	0,79	0,15	97,56	0,88	0,34	92,95	η
	3	8,25	1,07	89,00	8,94	2,39	82,33	δ
Edge top	1	0,18	-	98,06	0,20	-	92,90	η
	2	0,92	0,50	97,19	1,02	1,15	92,39	η
	3	9,49	0,87	87,66	10,20	1,94	80,46	δ
Edge bottom	1	-	0,03	98,71	-	0,08	94,94	η
	2	0,60	0,51	97,43	0,67	1,18	92,50	η
	3	9,33	0,99	87,86	10,05	2,20	80,88	δ

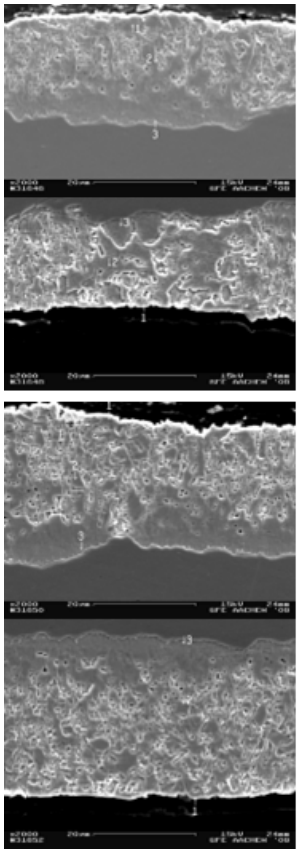
Fig. 17: SEM/EDX comparison between the middle and the edges for strip 1: 500°C, 20s

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224



Position on sample	EDX point	Elements in wt.%			Elements in at.%			Expected phase
		Fe	Al	Zn	Fe	Al	Zn	
Center top	1	7,83	0,49	89,77	8,49	1,11	83,16	δ
	2	8,51	0,79	89,22	9,29	1,79	83,26	δ
	3	13,54	0,18	83,18	14,14	0,39	74,19	δ + Γ1
Center bottom	1	0,39	0,14	97,98	0,43	0,32	93,43	η
	2	6,79	0,43	90,61	7,33	0,97	83,55	ζ / δ
	3	9,53	1,00	86,96	10,07	2,18	78,50	δ
Edge top	1	0,93	0,26	97,55	1,04	0,59	93,43	η
	2	6,64	0,73	90,74	7,19	1,63	83,99	ζ / δ
	3	12,62	0,22	84,08	13,19	0,48	75,08	δ + Γ1
Edge bottom	1	0,99	0,05	97,65	1,11	0,11	93,66	η
	2	1,52	0,17	97,24	1,71	0,39	93,67	η
	3	11,11	1,28	85,07	11,65	2,78	76,26	δ

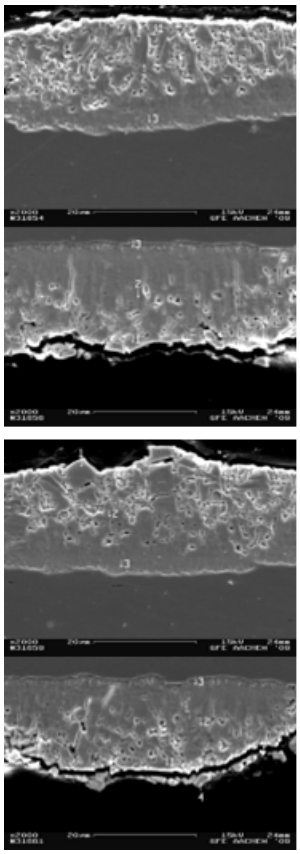
Fig. 18: SEM/EDX comparison between middle and edge for strip 2: 525°C, 20s



Position on sample	EDX point	Elements in wt.%			Elements in at.%			Expected phase
		Fe	Al	Zn	Fe	Al	Zn	
Center top	1	7,75	0,71	89,33	8,31	1,58	81,84	δ
	2	5,87	0,54	91,71	6,39	1,22	85,25	ζ
	3	13,86	0,21	82,38	14,28	0,44	72,51	δ + Γ1
Center bottom	1	0,54	0,22	97,94	0,60	0,51	93,78	η
	2	0,99	0,19	97,77	1,12	0,44	94,32	η
	3	8,60	1,07	87,73	9,06	2,33	79,01	δ
Edge top	1	0,16	0,16	98,10	0,18	0,37	93,32	η
	2	7,85	0,96	89,25	8,45	2,15	82,11	δ
	3	11,22	0,13	86,94	12,24	0,30	80,99	δ
Edge bottom	1	0,02	-	98,31	0,02	-	93,37	η
	2	2,09	-	96,20	2,31	-	90,98	η
	3	12,02	0,25	86,24	13,14	0,56	80,57	δ

Fig. 19: SEM/EDX comparison between middle and edge for strip 3: 500°C, 30s

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224



Position on sample	EDX point	Elements in wt.%			Elements in at.%			Expected phase
		Fe	Al	Zn	Fe	Al	Zn	
Center top	1	0,98	0,24	97,92	1,11	0,56	94,89	η
	2	6,67	0,63	90,83	7,24	1,42	84,26	ζ / δ
	3	13,16	0,44	82,73	13,49	0,94	72,45	δ + Γ1
Center bottom	1	8,79	0,27	89,14	9,58	0,62	82,98	δ
	2	9,05	0,41	89,02	9,91	0,93	83,33	δ
	3	12,14	0,35	85,18	12,94	0,77	77,57	δ
Edge top	1	6,89	0,26	91,44	7,62	0,59	86,34	δ (/ ζ)
	2	6,96	/	91,29	7,61	/	85,34	δ
	3	17,00	0,27	78,48	17,09	0,56	67,42	Γ1
Edge bottom	1	0,30	8,33	89,16	8,98	0,68	82,06	η
	2	8,75	2,10	87,01	9,22	4,59	78,33	δ
	3	14,26	0,21	82,97	15,09	0,46	75,00	δ + Γ1
	4	9,26	0,50	88,70	10,12	1,13	82,84	δ

Fig. 20: SEM/EDX comparison between the middle and the edges for strip 4: 525°C, 30s

The temperature difference between the middle and the edges is only about 5 – 10 °C, thus, a similar GA coating between the middle and the edges has been observed.
The inhomogeneous diffusion between both sample sides is due to the fact that the coating on one side is always slightly thicker than on the other side and/or because of a sample bending during Rhesca simulations.
It is recommended that the sample side with the thermocouple should be chosen as the sample side with the thicker coating for processing GA with the Trebel simulator.
Anyway, additional trials will be carried out (with pre-load to avoid sample bending effect for example) and also with IF steel. Although the strip temperature profile between the Zn-bath exit and the start of heating in the galvannealing furnace is considered to be a critical period for the control of nucleation and early growth of the Zn/Fe alloy phases with considerable influence on the course and final state of the subsequent alloying reaction, Mr. Strutzenberger (Voestalpine) underlined during the GAP meeting in May 2009 the fact that he experienced that results of GA simulations without and with intermediate liquid Zn solidification are fairly comparable.
Also, if this GA processing with intermediate solidification is followed for adhesively bonded joint testing, lap shear test specimen geometry should be modified and adjusted to Trebel simulator samples geometry.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Subtask 2.1: Selection of steel grades for galvannealing

The experimental study will first begin with an IF steel grade as discussed during the last GAP meeting in November 2008 before proceeding with an advanced high strength steel (AHSS) with a multiphase microstructure (DP steel grade would be of great interest for the GAP sponsors). In order to guarantee the industrial relevance of this project, the necessary materials should be provided by GAP sponsors.

From the literature study of Task 1, recommendations for the experimental study can be proposed. It would be interesting to:

- Investigate the substrate roughness influence,
- Investigate the influence of the nature and amount of the Γ/Γ1 phase at the substrate/coating interface,
- Consider the coating microstructure and phase make up instead of or additionally to the Fe composition dependence of the GA adhesion and shear strengths,
- Investigate which Fe content (low or high) should be targeted to reach improved shear strength in accordance with other coating properties (powdering…),
- Correlate the influence of AHSS alloying elements combination

The most important parameters influencing the galvanneal have to be investigated: substrate roughness, GA temperature and exposure time. Other experimental interests resulting from the survey concerning GAP sponsors requests and comments will also be considered after discussion.

A IF steel grade is helpful to verify that the well known GA results are reproducible.
Also, these steel grade is the most suited to study the influence of substrate roughness (important to differentiate between element segregation in grain boundaries from the roughness influence). Two different surface properties (rough vs. polished) and two different GA-temperatures (high vs. normal/low) should be used therefore. Polishing may change the element distribution at the surface, so a “smooth rolled at lab” material should be tried therefore, this possibility will be checked with IBF of RWTH Aachen University.

Ti ULC may have non recrystallized grains at the extreme surface. In that sense, Ti-Nb or Ti ULC with limited Ti excess may be better suited.
Si added IF steel are considered to be better for GA coating properties like lap shear strength as mentioned in the literature. Moreover, such Si-added variant is more reliable than conventional IF variant concerning ThyssenKrupp Steel (TKS) supplies.
About 10m² steel sheets were supplied from TKS (Mr. Norden). Material information and chemical composition is listed in Table 2.
The literature review pointed out that Ti-Nb-IF steels ensure higher shear strengths during lap shear test than Ti-IF steels and that the good GA coating adhesion can be improved with Si added Ti-Nb-IF steels. Thus, Si added Ti-Nb-IF steel was requested but Si added Ti-IF steel was supplied.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Material	Thickness	Supply condition	Delivery date
Si added Ti-IF (M3A42)	1,0 mm	CR, full hard	middle of March 2009

M3A42	C	Si	Mn	P	Cr	Mo	Ni	Al	Nb	Ti	B
wt%	<0,005	0,087	0,100	0,011	0,024	0,010	0,024	0,026	<0,001	0,063	<0,0005

Table 2: Material information and chemical composition of Si-added Ti-IF steel from TKS

The question during the GAP meeting in May 2009 was if we process with it or rather with another steel grade, like Ti-Nb IF as intended, because of Ti% influence on recrystallization. It was decided that another IF steel grade should be used as a reference material. A Ti-Nb IF steel without Si addition was suggested (typical Si-content is 0,025%, Mn% is also important).
Mr. Strutzenberger (Voestalpine) offered to supply some industrial “GI” IF material (like with Al% in Zn-bath as Al% used for GA) for GA simulations with intermediate liquid Zn solidification. But this type of processing can only be useful for investigations on IF steel (not for multiphase steels).
Additionally, IF steel in cold rolled full hard condition should be supplied for continuous GA simulations to investigate the substrate roughness influence.

Advanced high strength steel (AHSS) with a multiphase microstructure will also be integrated to the work program, since DP steel grade would be of great interest for the GAP sponsors. The AHSS grade will depend on GAP sponsors wishes and supplying possibilities, since the literature review didn’t pointed out specific conclusions on AHSS.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Task 3: Shear strength testing of adhesively bonded samples

Steel Selection

Two commercial DP600-GA steel grades were delivered by ILZRO sponsors. The GA coating is about 10µm thick for both steels and the microstructure was revealed using Kilpatrick etching. The chemical composition of these two steel grades has been identified by a spectroscopic analysis, shown in Fig. 21 and Fig. 22.

DP600 ThyssenKrupp (Sheet thickness: 0,8 mm)

DP600-GA	C	Si	Mn	P	Cr	Mo	Ni	Al	Nb	Ti	B
wt%	0,072	0,037	1,851	0,013	0,168	0,181	0,023	0,044	0,002	0,001	<0,0005

Fig. 21 Spectroscopic Analysis of DP600 ThyssenKrupp

DP600 USSteel (Sheet thickness 1,6 mm)

DP600-GA	C	Si	Mn	P	Cr	Mo	Ni	Al	Nb	Ti	B
wt%	0,089	0,169	1,254	0,012	0,124	0,207	0,023	0,032	<0,001	0,002	<0,0005

Fig. 22 Spectroscopic Analysis of DP600 (US Steel)

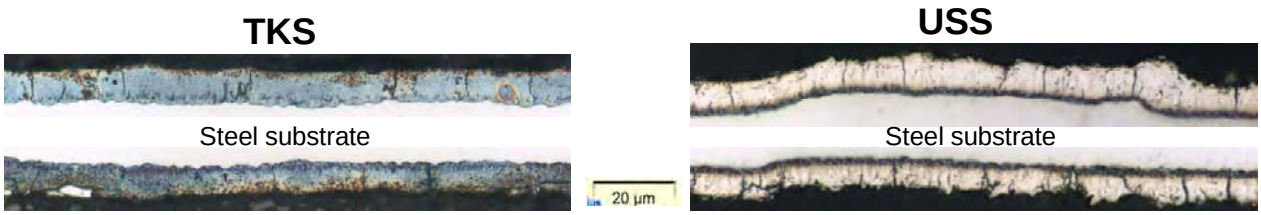


Fig. 23: Cross-sections of DP600-GA coating under light optical microscope

The surface topography and roughness of both steels were studied with a confocal microscope at IEHK.
The equipment from the company Nanofocus is a variant of a light microscope. Several applications are possible with this tool:

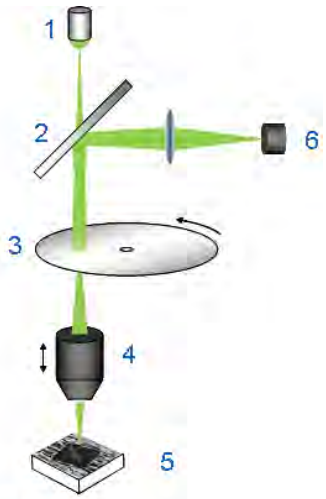
- Geometric measurements
- Surface technology: roughness measurement, characterisation of functional surfaces…
- Characterization of wear and degradation
- Microstructural description: perlite interlamellar spacing measurement, visualization of TWIP mechanical twins structure and identification of different phases due to different etching behaviour

The equipment description is presented in Fig. 24. The measurement of the provided steel sheets is shown in Fig. 25.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

CCD resolution [pixels] x [pixels]	512 x 512
Lens	160-S
Magnification	100 x
Measuring field [µm] x [µm]	160 x 160
z-resolution [nm]	1*
x,y-resolution [nm]	0,31

* Optical limitations (noise)



1: led light source
2: beam splitter
3: pinhole filter (Nipkow disk)
4: lens (adjustable by piezo quartz)
5: sample
6: CCD camera

Fig. 24: Confocal Microscope Technical Data

Statistical roughness evaluation (cut off: 0,25mm)

Steel	**Ra** [µm]	**Rz** [µm]	**Rq** [µm]	**RPc** [1/mm]	**Rmax** [µm]
TKS *DP600-GA*	0,942	6,83	1,24	19,5	7,72
USS *DP600-GA*	0,638	4,51	0,821	17,5	5,03

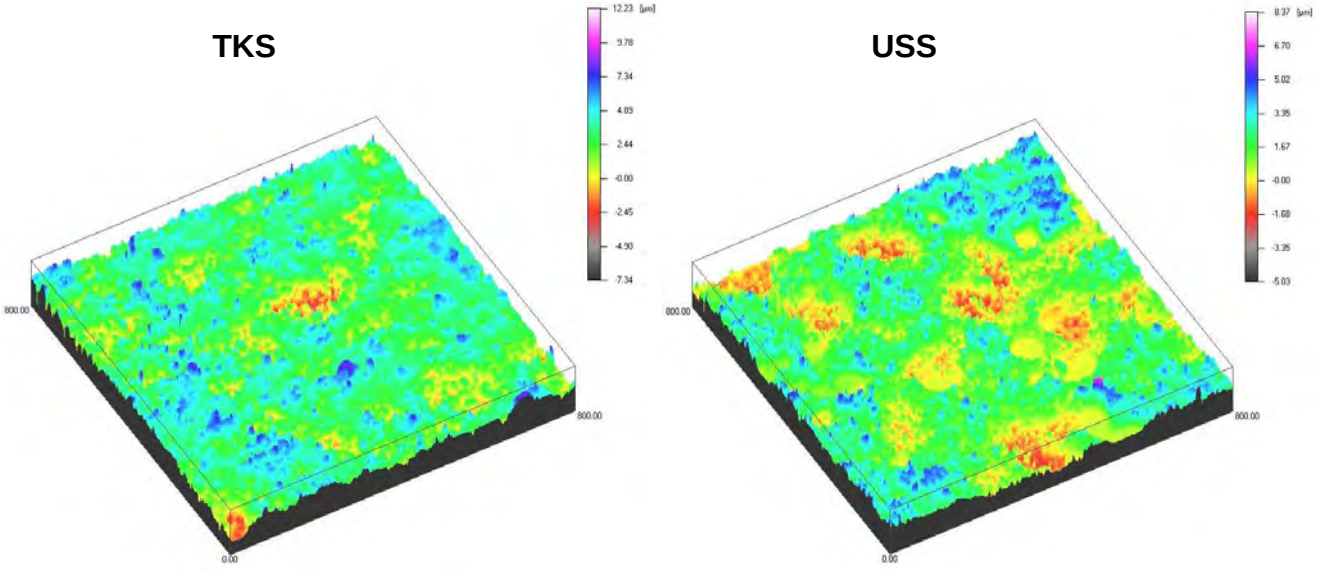


Fig. 25: Roughness measurement of the provided steels

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Subtask 3.1: Characterization of adhesives

Selection of adhesives

To identify an adequate adhesive for bonding galvannealed steel a lot of influencing factors have to be considered. Next to the mechanical properties, the thermal properties and the rheological properties, especially the chemical properties which are significantly responsible for the development of adhesive forces between adhesive and substrate, have to be analyzed.
The surface of galvannealed steel mainly consists of zinchydroxide and zinccarbonate layers, hence the used adhesive has to be able to develop sufficient adhesive forces to these surfaces.
Epoxy resins show a high potential for adhesively bonding galvannealed steel based on their capability of developing chemical complex bonds with the zinc rich surface.
In consultation with the leading adhesive producers 5 epoxy resins have been chosen for preliminary testing:

Terokal 5071 is a heat curing one component adhesive based on epoxy resin. It is designed to resist high peel forces and impact peel energies. It is mainly used in the automotive industry, especially in the body shop.

Araldit AW116 is a room temperature curing universal two-component adhesive based on epoxy resin. Strong adhesive bonds and durability qualifies this adhesive for a lot of joining materials.

Betamate 1496 V is a heat curing, one component, epoxy based adhesive especially developed for the body shop. An excellent adhesion to coated steels and pre-treated aluminium qualify this adhesive for the joining task.

Delo Monopox 1196 is a heat curing, one component adhesive based on epoxy resin. It is filled with alumina fillers to increase the mechanical properties especially under the influence of high temperatures.

3M 7036 is a heat curing, two component, epoxy based adhesive designed for the adhesive bonding of steel. The high tolerance for deep-drawing oil and the good response time for induction curing characterize this adhesive.

Since the chemical, mechanical and technical details delivered by the adhesive manufacturers are incomplete and inconsistent regarding testing approach, material, pre-treatment and different charges of the adhesive differ to a small extent from one another, measurements of the decisive influencing factors have to be done with equal test set-ups.

Considering the aim of the project, being the correlation of the parameters used in the hot-dip galvannealing process and the adhesively bonded samples with the mechanical and chemical properties of the whole composite, a characterization of the adhesives recommended in task 1 is necessary to determine the relevant influencing variables. Since the manufacturer's data is often incomplete and dependent on the actual production batch, a characterization of every adhesive before utilisation is needed.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Mechanical characterization

Shear tests

Besides the characterization of the adhesive properties, e.g. Young's modulus, Viscosity, Glas-transistion temperature, a simple shear test on commercial produced galvannealed steel qualifies the performance of the respective adhesive in an applied loading.

Sample preparation:
According to DIN EN 1465 shear test specimen were produced with the following parameters:

- Pre-Treatment:: Degreasing with Isopropanol
- Adhesive layer thickness: 200µm +/-20µm
- Curing: According to the respective adhesive technical data sheet
- Overlap length: 12,5mm and 18mm

A PTFE Layer is applied to guarantee a reproducible adhesive layer area. The adhesive layer thickness is adjusted by glass balls with a diameter of 200µm. To ensure a reproducible specimen preparation a clamping fixture has been used, Fig. 26.

Test Set-Up:
After curing the adhesively bonded specimen according to their technical data sheets, testing has been carried out on a 100kN tensile testing machine, Fig. 26 right.

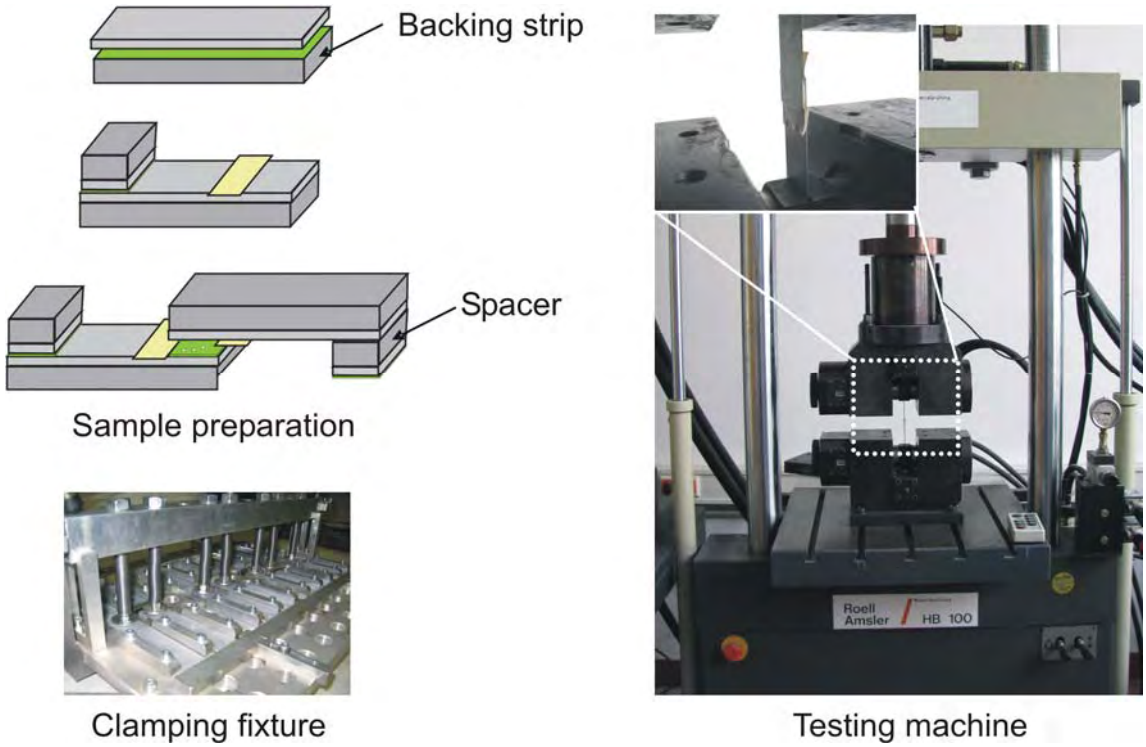


Fig. 26: Shear Test; Left: Sample Preparation; Right: Testing Equipment

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Testing Parameters:
Testing Speed: 1mm/min
Data Acquisition: Load, Piston stroke, time
Sampling rate: 50 Hz

Analysis:
The tested specimen have been analysed regarding their stress-strain behaviour and fracture surface. Since only the piston stroke has been measured, the strain behaviour can only be compared to the other tested adhesives. In the following the 12,5mm overlapped specimen will be referred to as short specimen while the 18mm overlapped specimen will be referred to as long specimen. The DP600 ThyssenKrupp steel will be referred to as TK600 while the DP600 USSteel steel will be referred to as US600.

Terokal 5071

The short TK600 specimen showed a complete cohesive failure in the adhesive at a stress level of about 25 MPa which correlates with the manufacturer's tensile test specifications of the bulk material (28MPa).

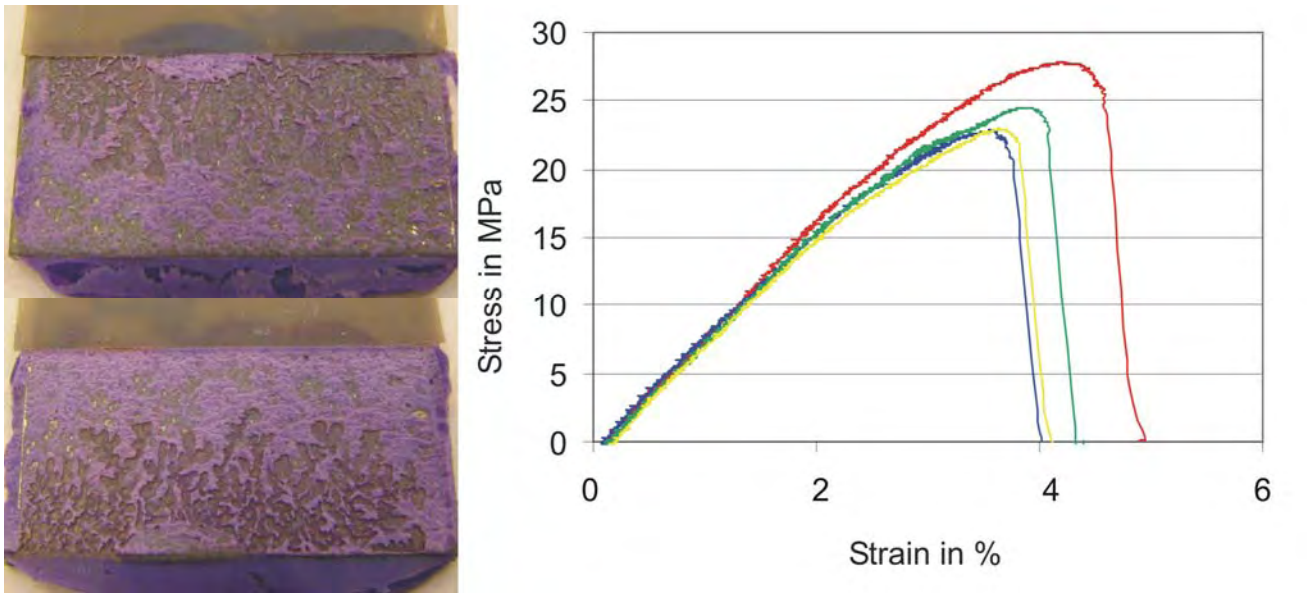


Fig. 27: Fracture surface and Stress-Strain Diagram of Terokal 5071 12,5mm (TK600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

The long TK600 specimen showed a mainly cohesive failure of the adhesive with partial failures in the galvanneal coating. Whether the galvannealed coating fails adhesively at the base material/galvanneal coating interface or cohesively inside the galvanneal coating cannot be told without an adequate analyzing tool.

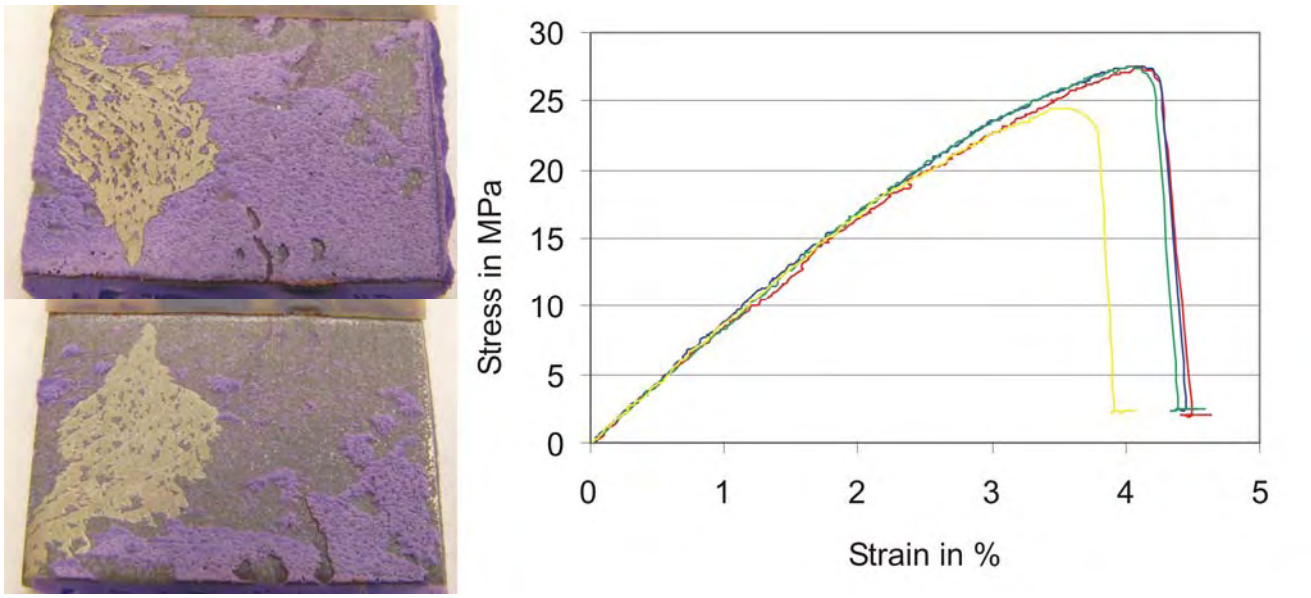


Fig. 28: Fracture surface and Stress-Strain Diagram of Terokal 5071 18mm (TK600)

The short US600 steel sheets show a mixed fracture surface, meaning there is no consistent failure in one interface identifiable.

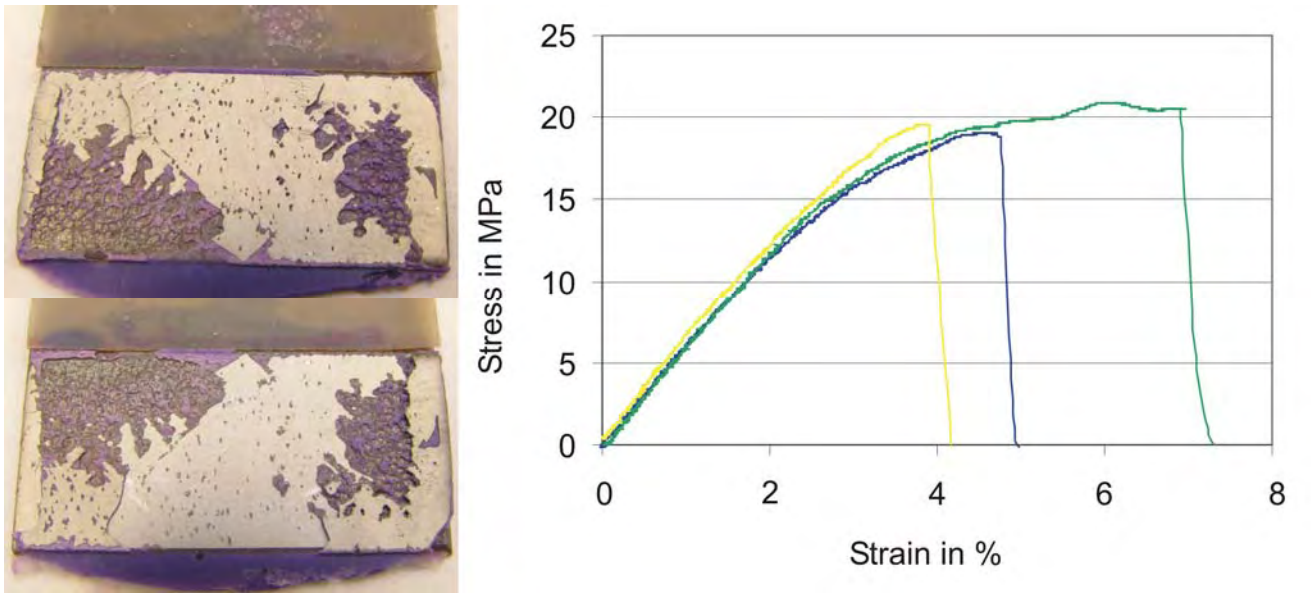


Fig. 29: Fracture surface and Stress-Strain Diagram of Terokal 5071 12,5mm (US600)

In the long US600 specimen this fracture behaviour is even more recognizable. At a stress level of about 17MPa the thinner US600 steel deforms ductile. This explains the higher breaking strain of the thinner US600 sheets.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

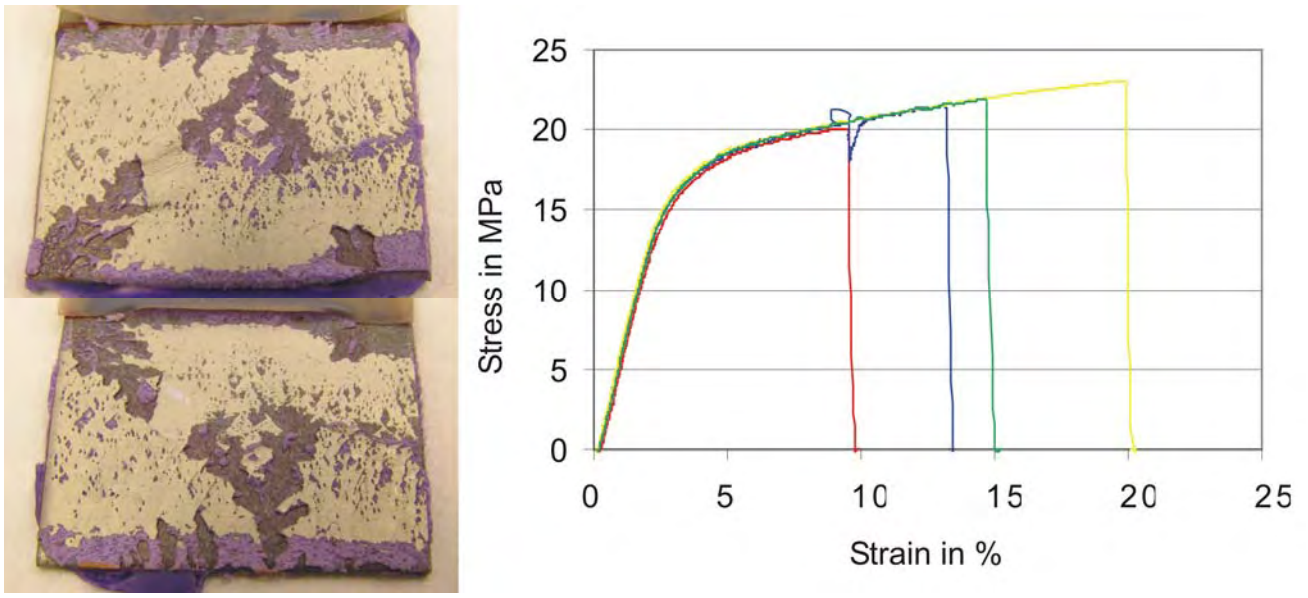


Fig. 30: Fracture surface and Stress-Strain Diagram of Terokal 5071 18mm (US600)

Betamate 1496 V

Both, the long and the short TK600 steel sheets show a mere cohesive failure in the adhesive when bonding with the Betamate 1496 V adhesive. Stress levels average at about 24 MPa. Since the Youngs modulus and the Shear strength of the Terokal 5071 and the Betamate 1496V bulk material is almost identical, the breaking strain of the shear tested specimen are comparable.

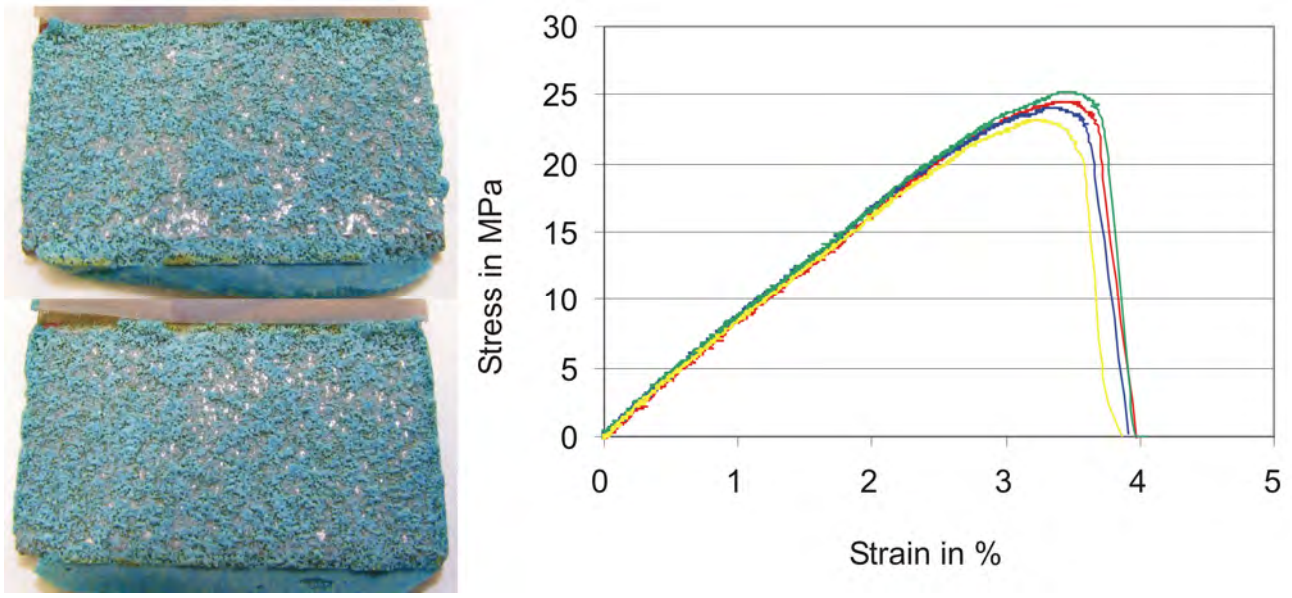


Fig. 31: Fracture surface and Stress-Strain Diagram of Betamate 1496 V 12,5mm (TK600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

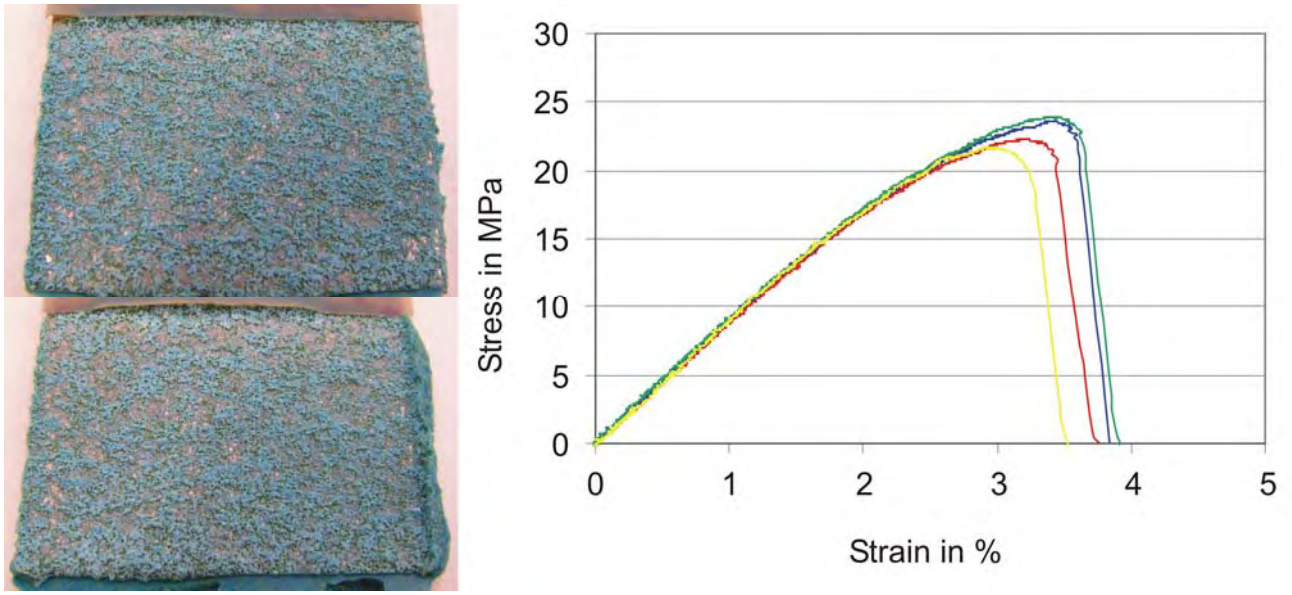


Fig. 32: Fracture surface and Stress-Strain Diagram of Betamate 1496 V 18mm (TK600)

The US600 samples show a mainly cohesive failure with some delamination of the galvannealed coating. The short US600 specimen show a similar Stress-Strain behaviour to the TK600 steel while the long specimen show a distinctive strain behaviour which is again caused by ductile steel deformation and induced at stress levels of about 17MPa. The strain extension correlates with the reaching of the steels yield stress and presumably initiates the failure in the longer specimen while the short shear test specimen fail nearly at stress levels of the bulk adhesive material. Failure mechanism presumably is a mixture of excession of the adhesive's tensile strength and initiation of the steels ductile deformation.

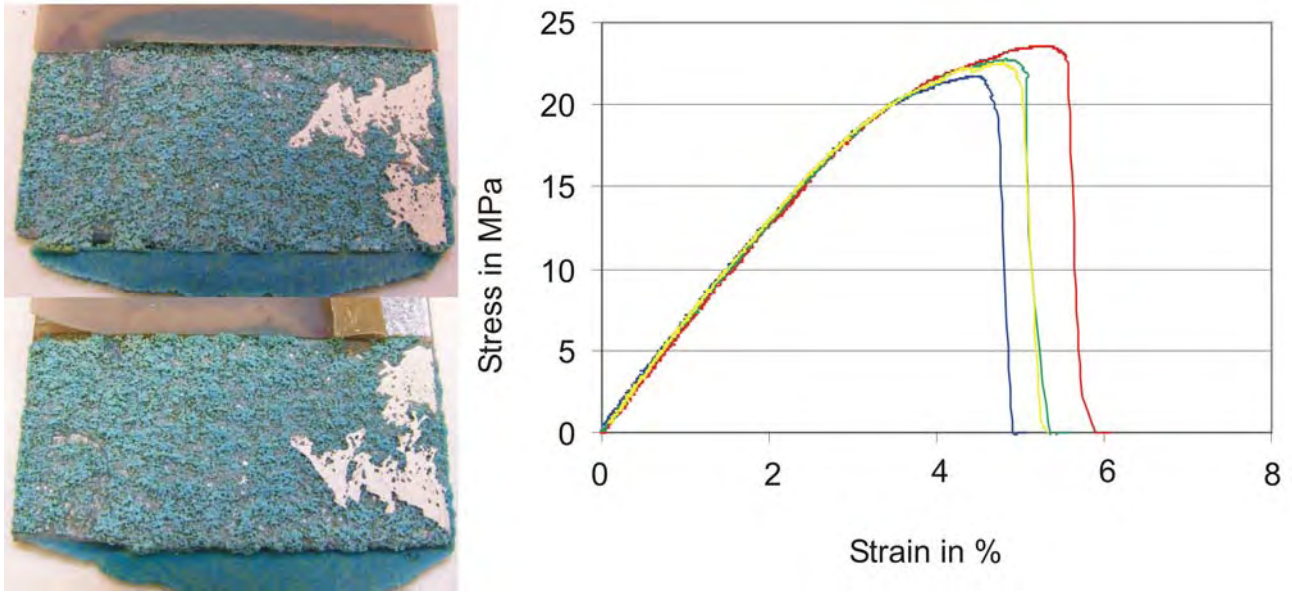


Fig. 33: Fracture surface and Stress-Strain Diagram of Betamate 1496 V 12,5 (US600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

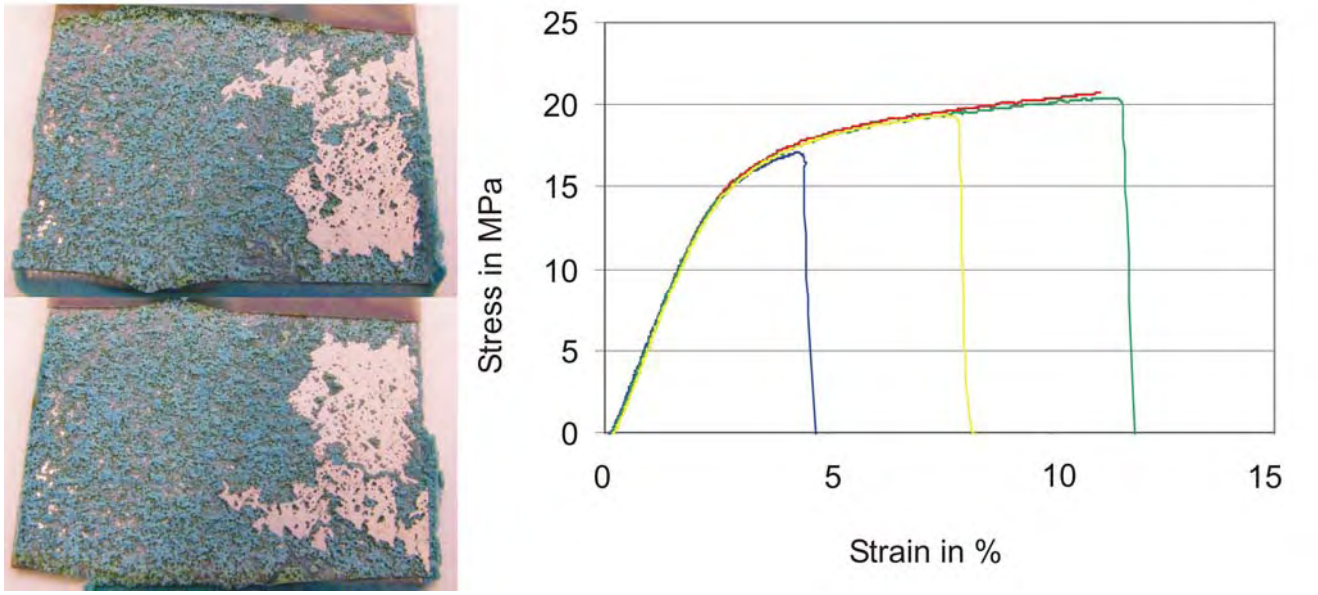


Fig. 34: Fracture surface and Stress-Strain Diagram of Betamate 1496 V 18mm (US600)

Monopox 1196

The with Alumina particles filled Monopox 1196 shows together with the Terokal 5071 adhesive the highest shear strength of the selected adhesives. According to the manufacturers' data the mean breaking elongation of the adhesive is noticeable in a sudden failure without a distinctive strain behaviour. After an optical analysis the short specimens seem to fail cohesively in the adhesive. When comprising the manufacturer's data, especially the tensile strength of the bulk material, the stress level should be higher at a complete cohesive failure. An explanation could be the asymmetric application of force caused by the simple shear strength testing method or an incomplete adhesive curing.

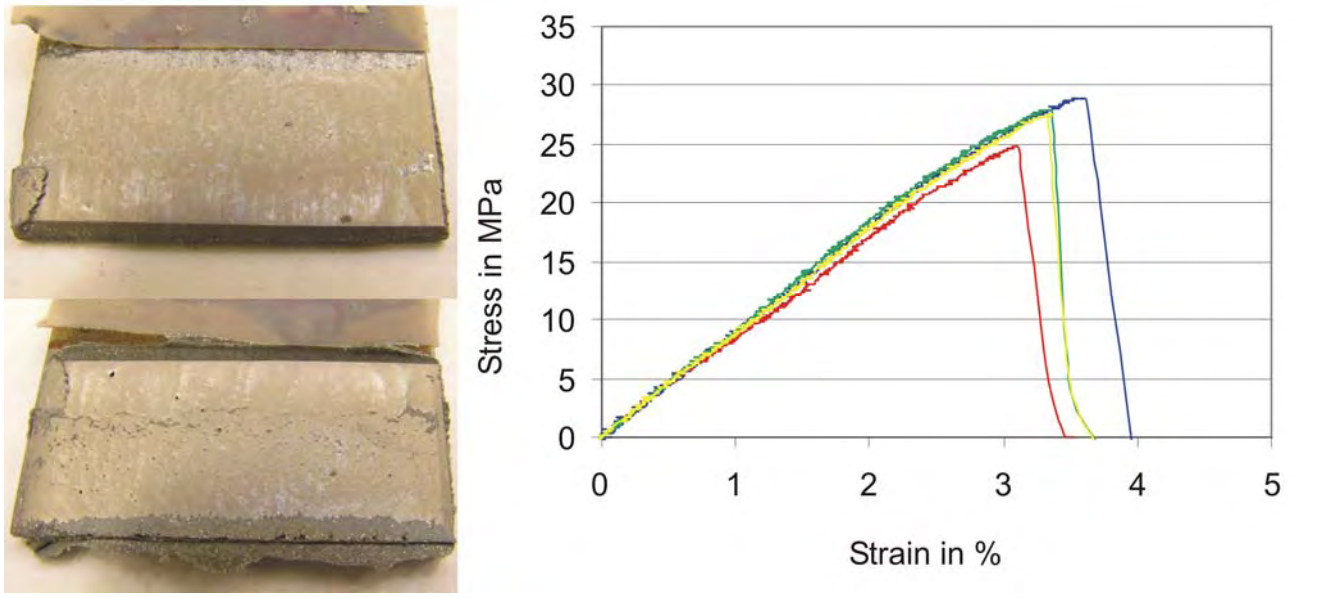


Fig. 35: Fracture surface and Stress-Strain Diagram of Monopox 1196 12,5mm (TK600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

The long specimens show a mixed fracture pattern. The failure appears to be initiated by the higher stress levels at the edges of the adhesively bonded area.

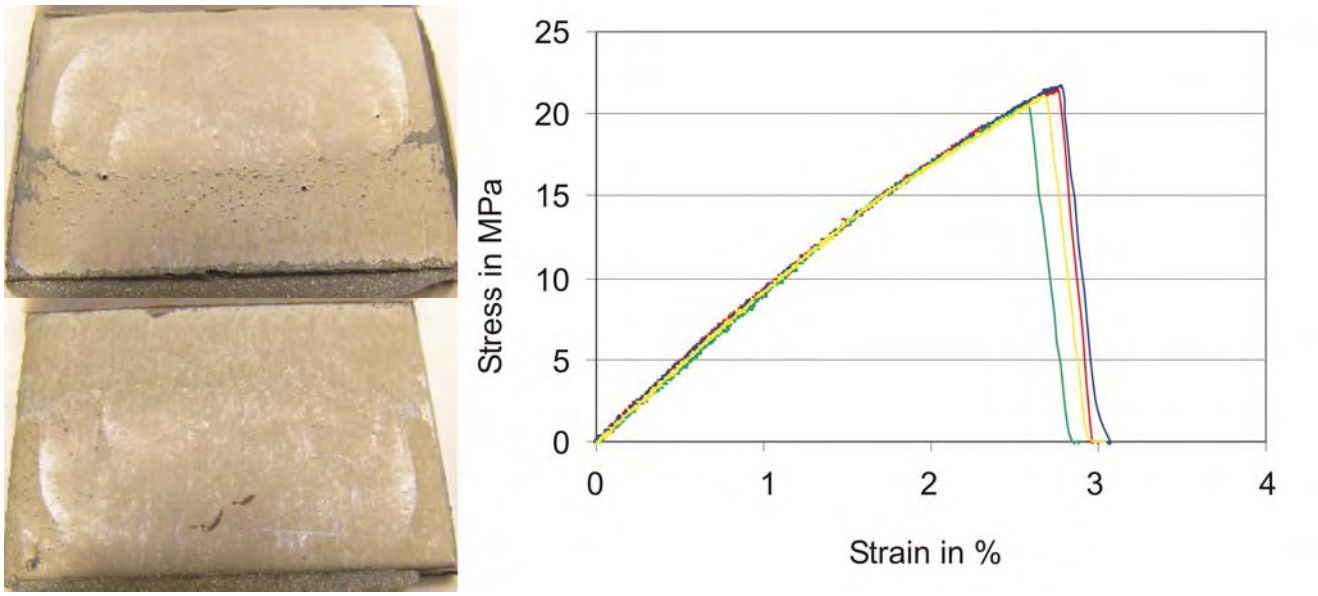


Fig. 36: Fracture surface and Stress-Strain Diagram of Monopox 1196 18mm (TK600)

Both, the short and the long specimen of the US600 steel showed an estimated complete adhesive failure in the base material/Galvannealed Coating interface layer. Again this can be correlated with the beginning plastic deformation of the base material.

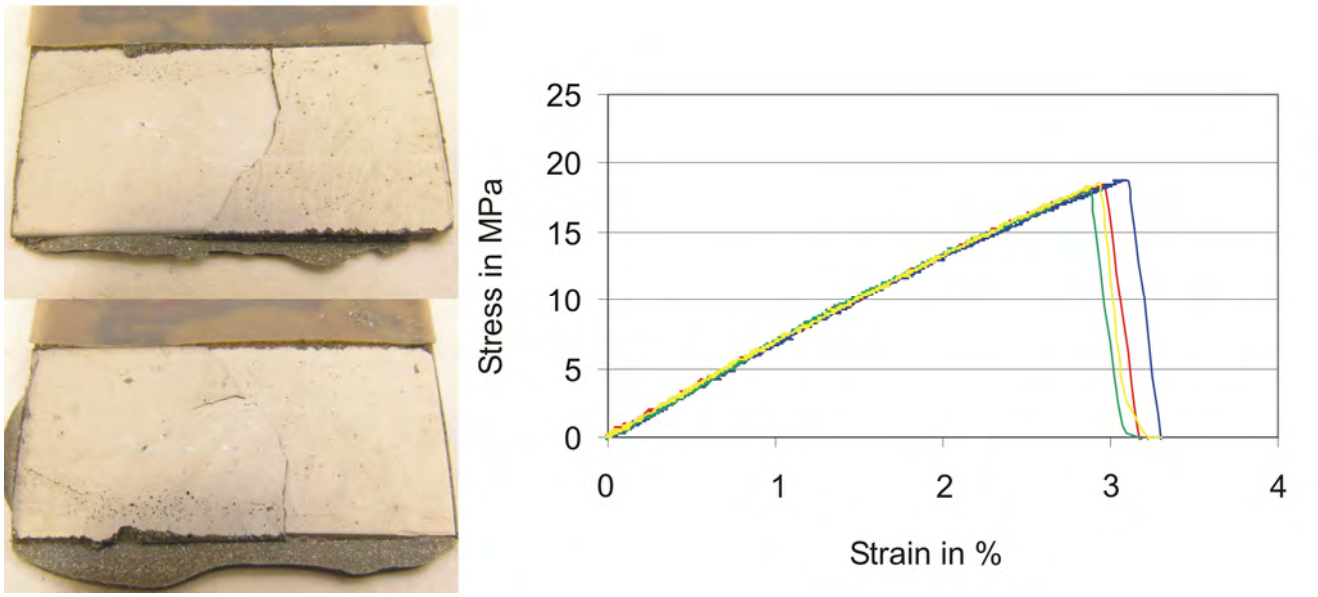


Fig. 37: Fracture surface and Stress-Strain diagram of Monopox 1196 12,5mm (US600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

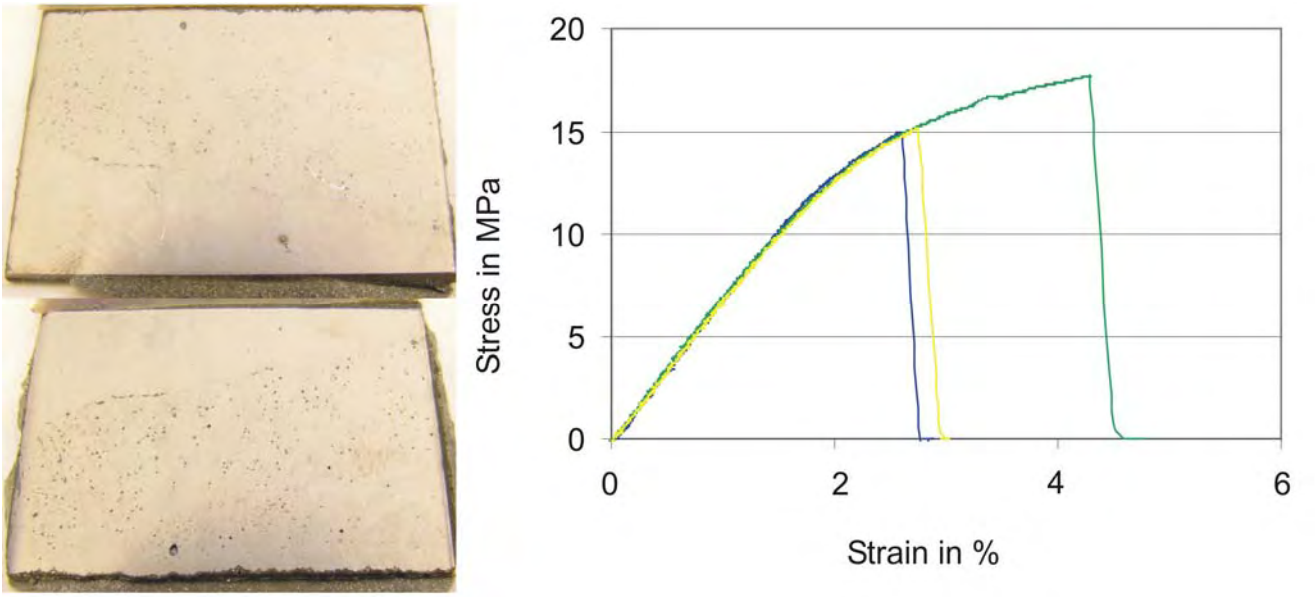


Fig. 38: Fracture surface and Stress-Strain Diagram of Monopox 1196 18mm (US600)

Araldit AW 116

The TK600 specimens that were adhesively bonded by the 2-component adhesive curing at room temperature show a mixed fracture pattern when using the short overlap length. Presumably the failure initiates at the edges of the bonded area. Furthermore it appears that the adhesive has adhesively failed in some parts of the fracture area. EDX scans could qualify this assumption. The long specimens show a similar fracture pattern as well as almost the same Stress-Strain diagram.

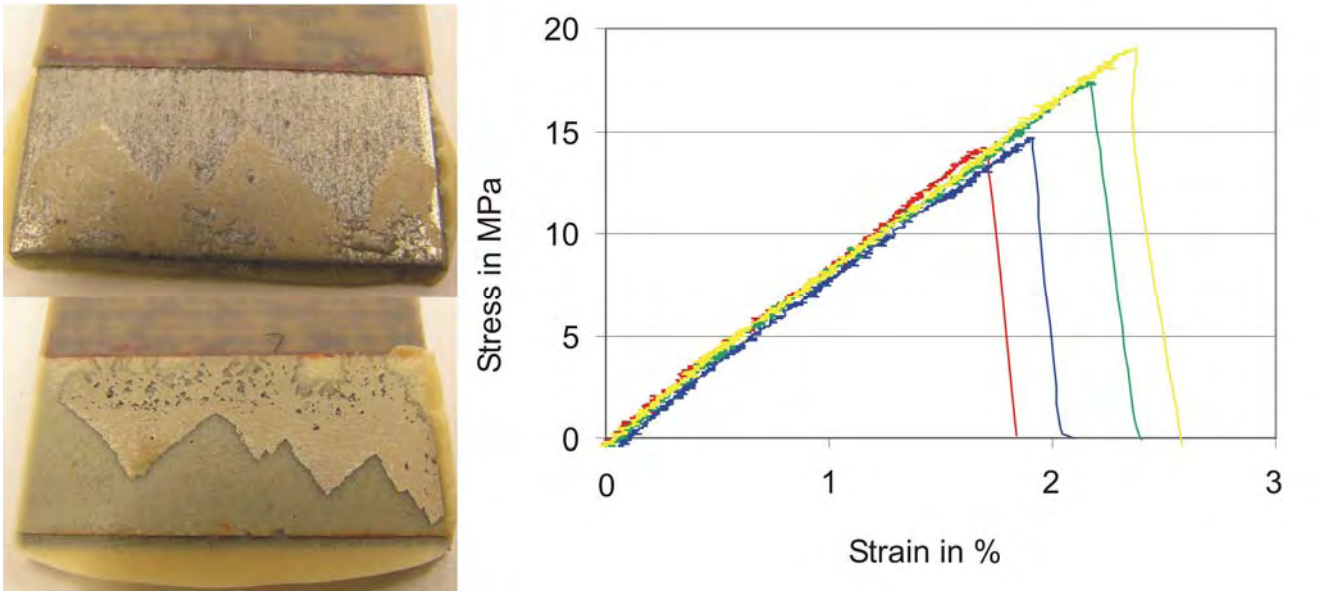


Fig. 39: Fracture surface and Stress-Strain Diagram of Araldite 116 12,5mm (TK600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

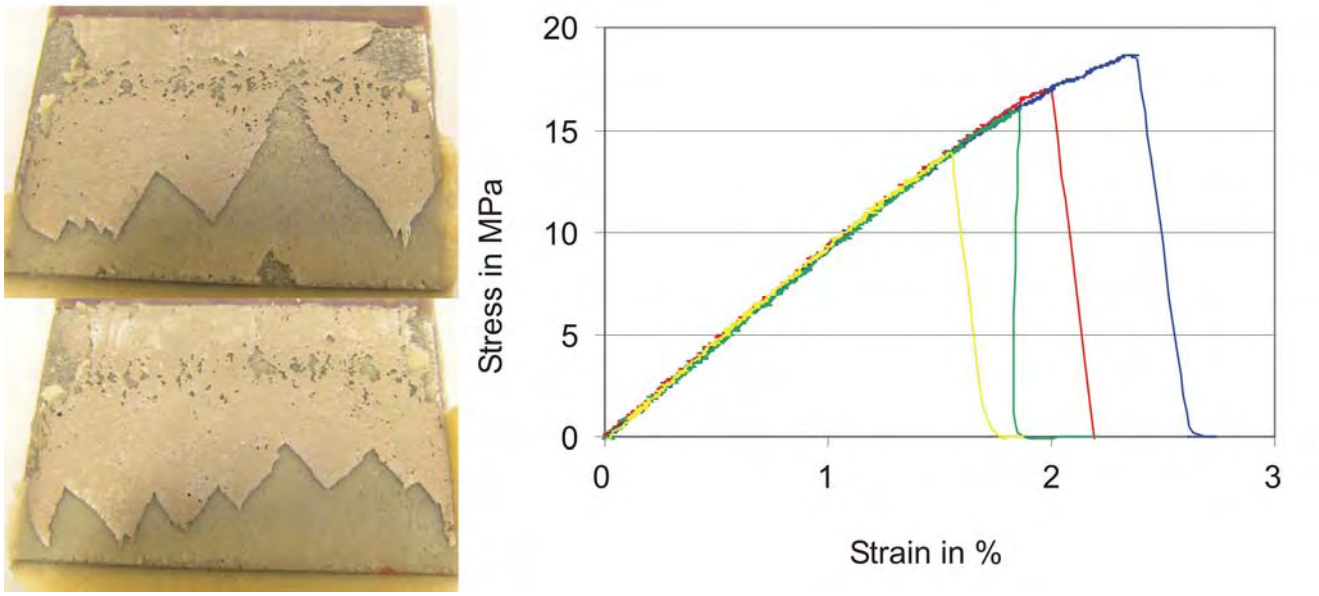


Fig. 40: Fracture surface and Stress-Strain Diagram of Araldite 116 18mm (TK600)

The US600 specimens show a different fracture pattern. Caused by the plastic deformation of the base material the fracture interphase is mixed with both, adhesive failures between the base material and the galvannealed coating and cohesive failures in the adhesive bulk material. Stress peaks and the higher plastic deformation of the steel sheet enhance this problem.

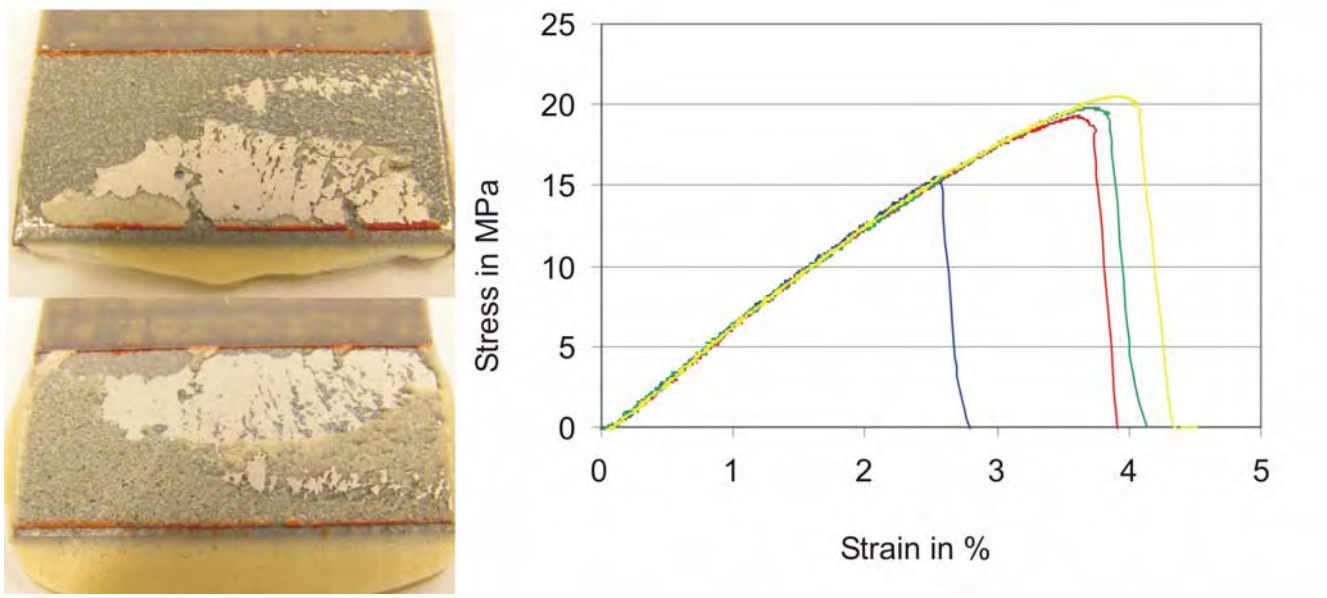


Fig. 41: Fracture surface and Stress-Strain Diagram of Araldite 116 12,5mm (US600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

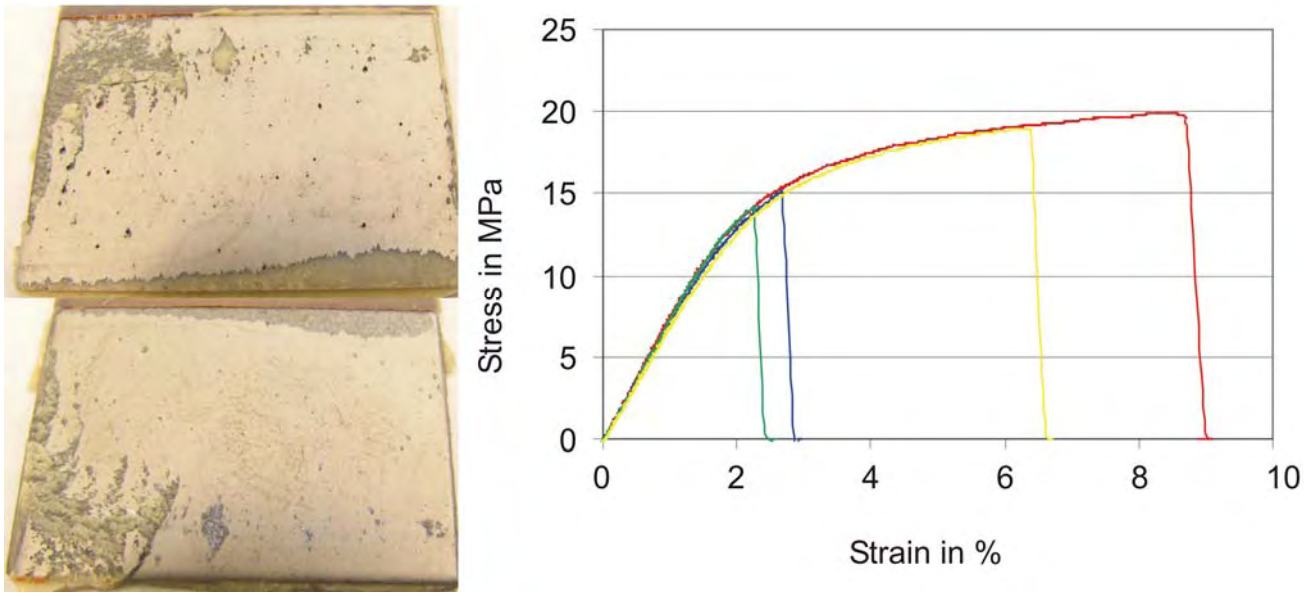


Fig. 42: Fracture surface and Stress-Strain Diagram of Araldite 116 18mm (US600)

3M

The 3M adhesives fracture plane is almost identical to the fracture planes of Terokal. The adhesive fails cohesively with the TK600 steel and short overlap length reaching Stress levels of 25MPa. With a longer overlap length, higher stress peaks provoke a premature failure of the adhesive. Consequently achievable stress levels decrease.

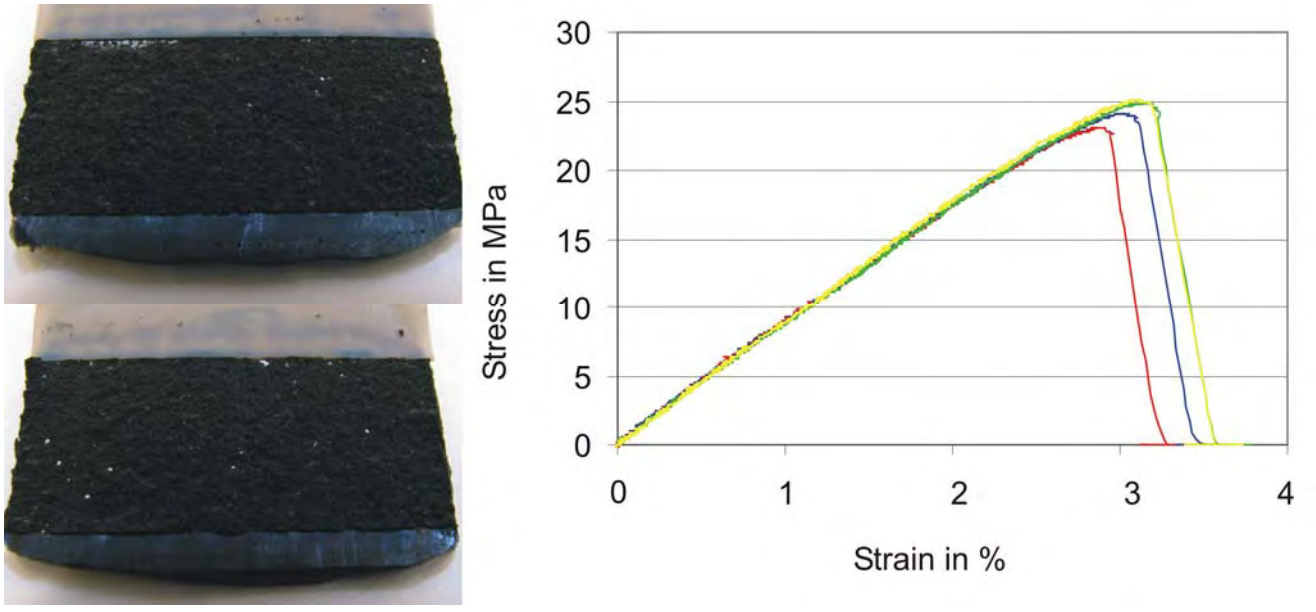


Fig. 43: Fracture surface and Stress-Strain Diagram of 3M 12,5 mm (TK600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

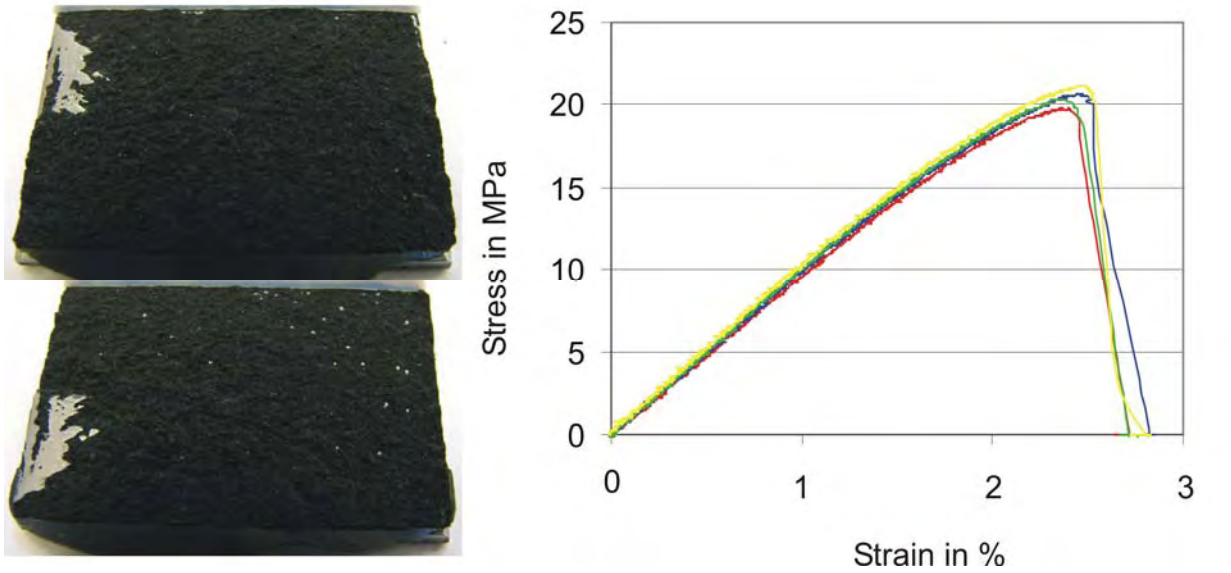


Fig. 44: Fracture surface and Stress-Strain Diagram of 3M 18mm (TK600)

The adhesively bonded US600 steel sheets again fail when reaching the steel yield stress resulting in a complex stress condition.

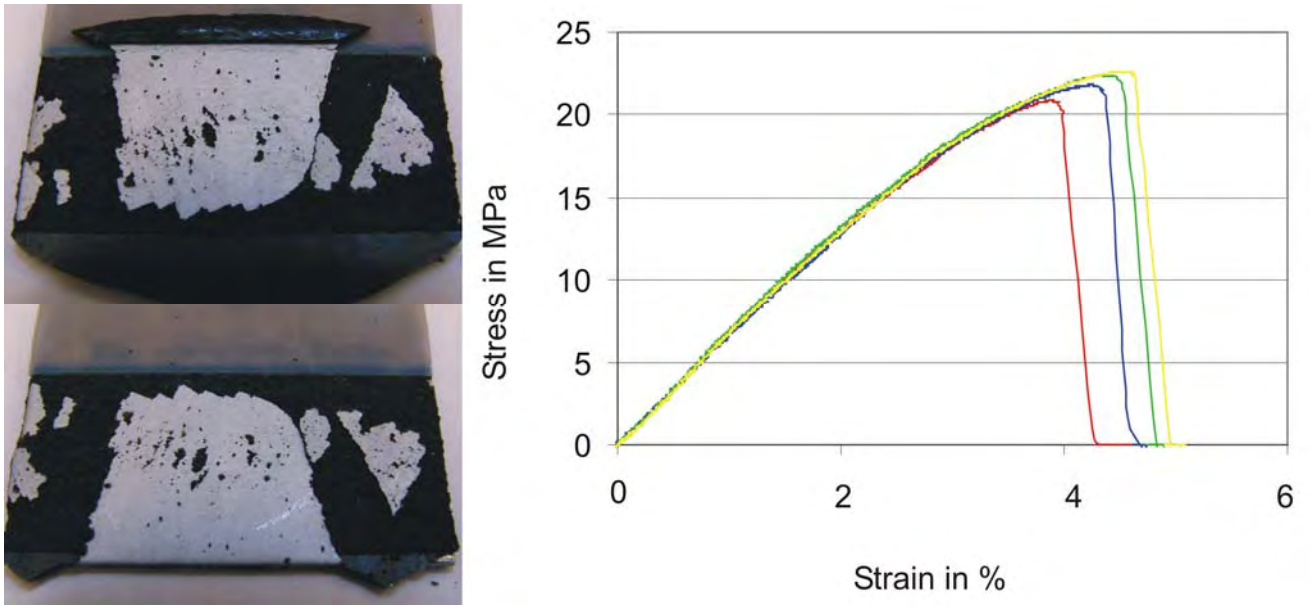


Fig. 45: Fracture surface and Stress-Strain diagram of 3M 12,5mm (US600)

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

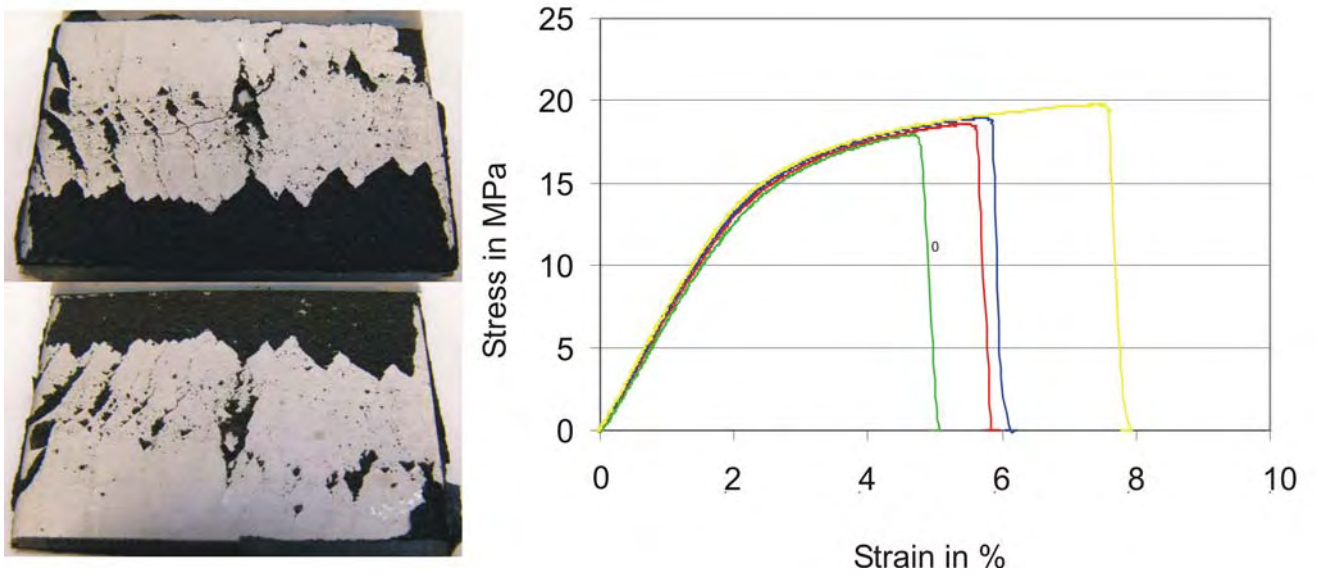


Fig. 46: Fracture surface and Stress-Strain diagram of 3M 18mm (US600)

Influence of the adhesives curing temperature

In this study thermal curing adhesives have been used. Curing temperatures vary between 100°C and 180°C. To identify whether the curing temperature alters the galvannealed steel sheets regarding stress relief, which could influence the testing results of the shear tests, several steel sheets were tempered with 180°C for one hour. After that a room temperature curing adhesive has been used to adhesively bond the tempered steel sheets.
Fig. 47 shows that both the stress/strain diagram and the fracture patterns are not altered by the steels tempering at 180°C, so that for our test results the adhesive curing temperature does not have to be considered.

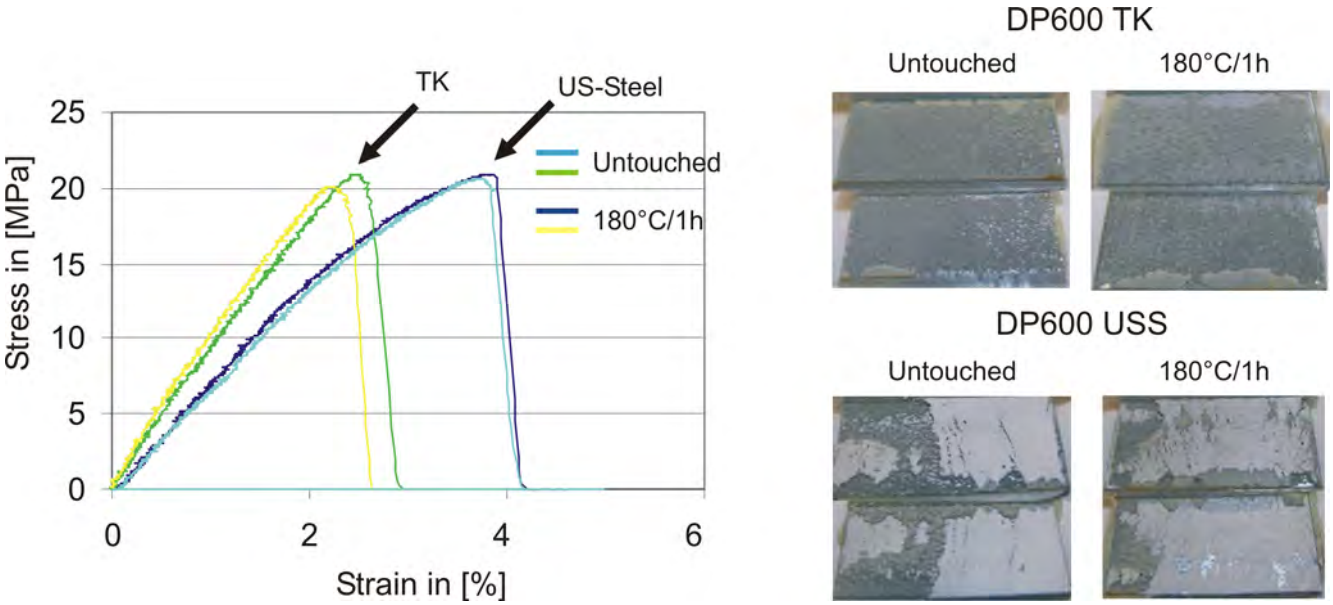


Fig. 47: Influence of the curing temperature

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Backed Up Shear Test

The standardized shear test is a simple method to compare different adhesives. Nevertheless, to quantify the resistance to shear strength of adhesively bonded galvannealed steels it is important to eliminate all other influencing factors.
Due to the complex stress condition when reaching the steels yield stress together with the asymmetric force transmission a comparison between the different adhesives otherwise is not sufficiently possible, hence a modification of the simple shear test is necessary.

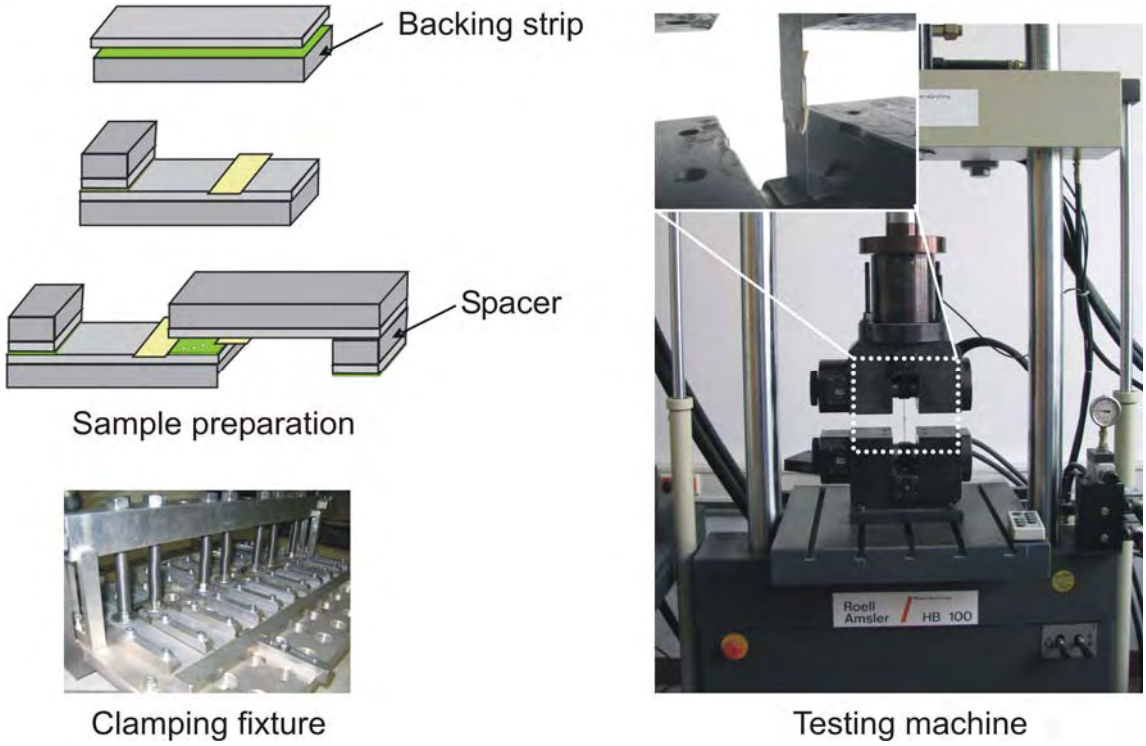


Fig. 48: Backed Up Shear Test US Steel; Left: Sample preparation; Right: Testing equipment

To eliminate the interference factors the test setup has been modified. Spacers to guarantee a centric load transmission are adhesively bonded on both steel sheets. Furthermore a backing strip is applied to stiffen the steel sheets, so that the steels yield stress does not influence the test results.
The stress/strain diagram and the breaking patterns of the Backed Up Shear test for the USS steel sheets are shown in Fig. 49.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

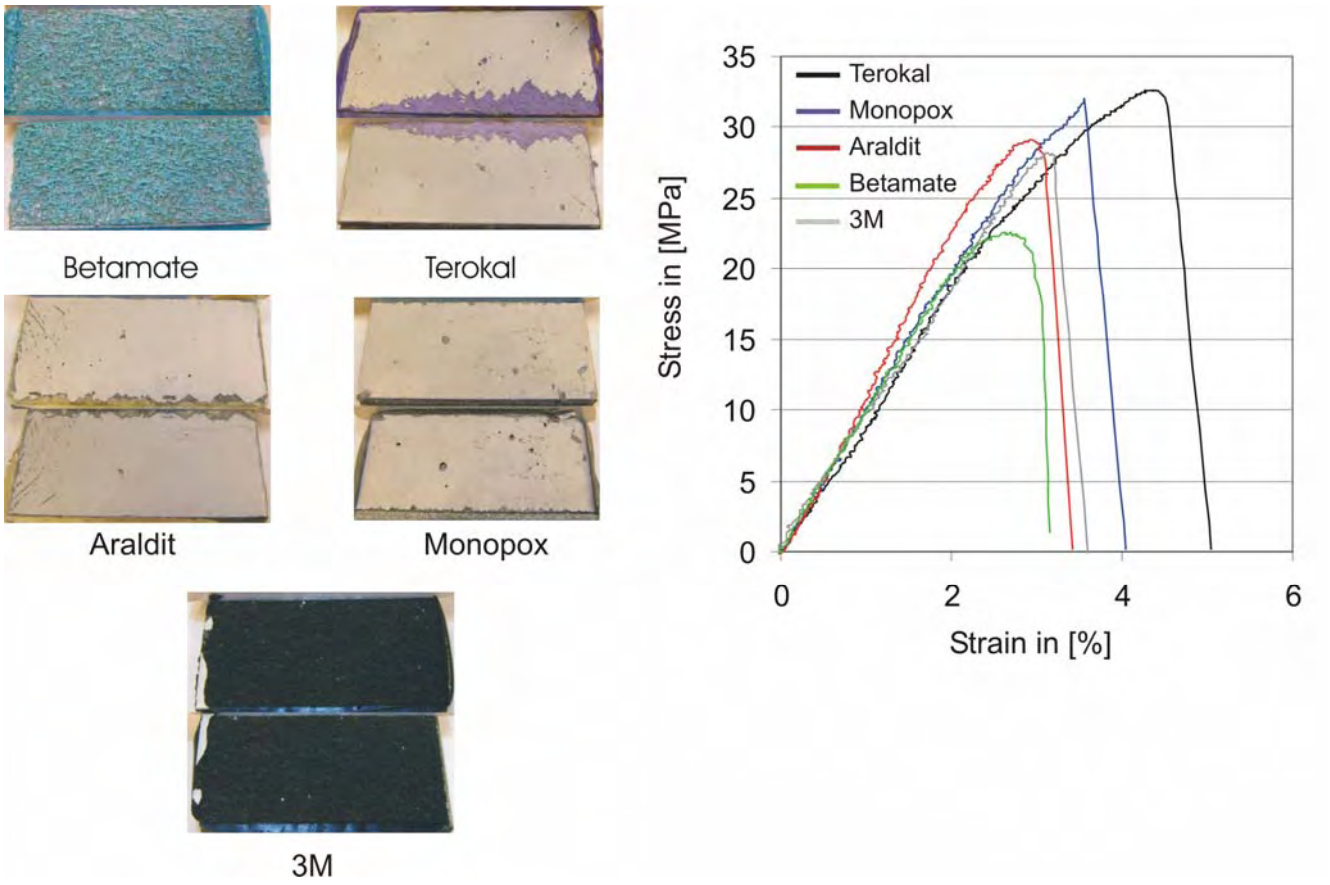


Fig. 49: Backed Up Shear test results for US600

In contrast to the simple shear test, specimen that are adhesively bonded with the Monopox, Terokal or Araldit adhesive obviously show a complete adhesive failure at the coating/Base material interface while Betamate and 3M fail cohesively in the adhesive. The premature failure of the adhesives in the simple shear test is likely caused by stress peaks resulting from the asymmetric load transmission. The homogenous breaking pattern is a result of the elimination of the steels plastic deformation during the shear test.
Consequently, when using steel sheets thinner 1mm, the backed up test setup has to be used. Furthermore for thicker steel sheets spacer have to be applied so that the adhesive does not fail prematurely.

Fracture Pattern analysis
Photo-optical analysis of the fracture pattern allow for a differentiation whether the specimen fail cohesively in the adhesive or adhesively at the adhesive/coating interphase. However, if the specimen fail in the GA coating a photo-optical analysis to discern in which phase of the coating the specimen failed is not sufficient because of the small dimensions.
Since the different phases vary in their chemical composition, EDX scans of the fracture patterns were made to qualify this analysis tool.
When differentiating the base material from the gamma phase is easily possible since the chemical composition of these two phases is very different, an analysis of two phases inside the coating is uneven harder.
To identify Zinc with EDX, the electron beam voltage has to be over 18keV, which results in a penetration depth of 2-3µm in the substrate. Since some phases in the galvannealed coating

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

might have thicknesses in these dimensions and since the fracture might proceed near to the interface of two phases, the electrons might have stimulated deeper located layers, Fig. 50.

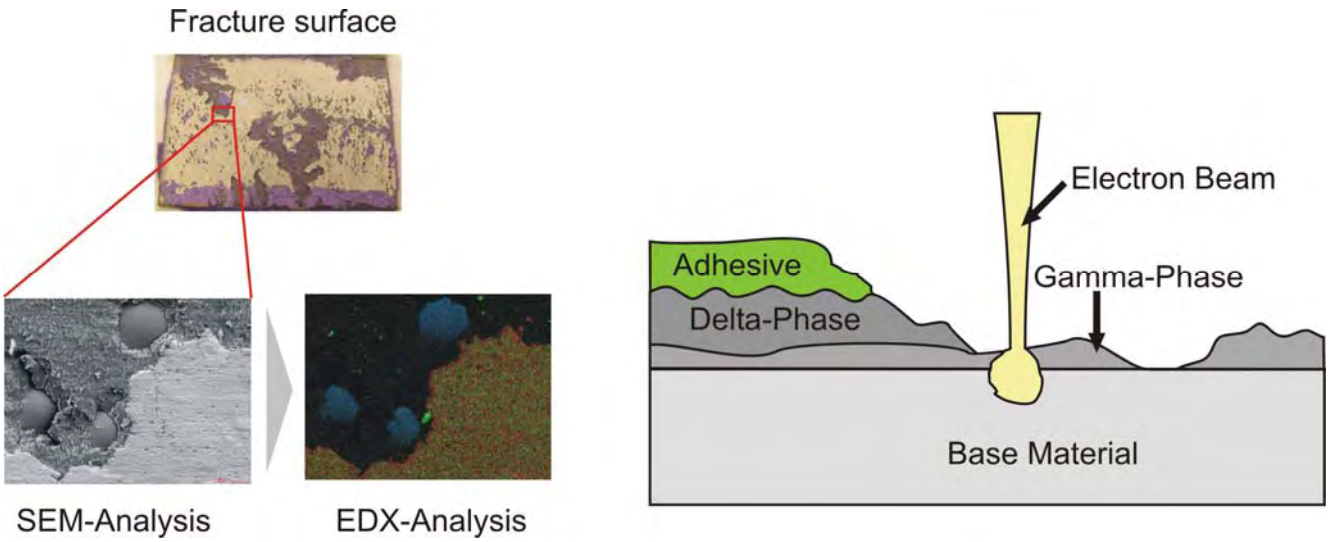


Fig. 50: EDX Analysis

Furthermore due to the fracture patterns dimensions an analysis of the whole fracture area is very complex.
Consequently, only spot tests are possible to identify failure mechanism and even then the results have to be treated carefully since deeper located layers might have been stimulated.

Peel Test

To qualify the adhesives adhesion strength to the galvannealed coating peel tests have been carried out. For this test two steel sheets of 200mmx25mm are adhesively bonded with an adhesive layer area of 150mmx25mm. The non bonded endings are bended in a 90° angle, clamped in a tensile testing machine and pulled apart with a speed of 100mm/min.
The test setup is shown in Fig. 51.

The testing results in Fig. 52 show, that all tested adhesives fail cohesively in the adhesive. Hence the adhesion to the galvannealed coating is sufficient, so that the adhesion to the zinc surface does not need to be improved during our project. Nevertheless it is shown that the Terokal adhesive shows a high potential for this mechanical load, failing at the highest stress/strain levels.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

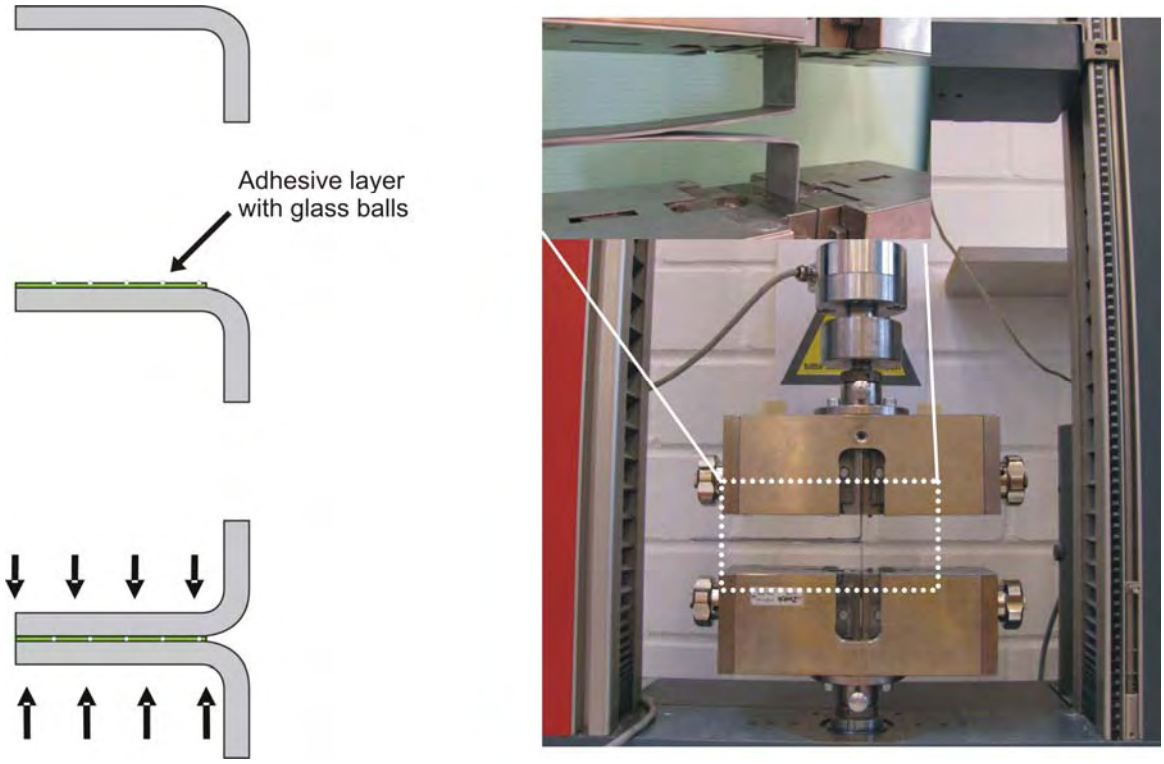


Fig. 51: Peel Test; Specimen preparation and test setup

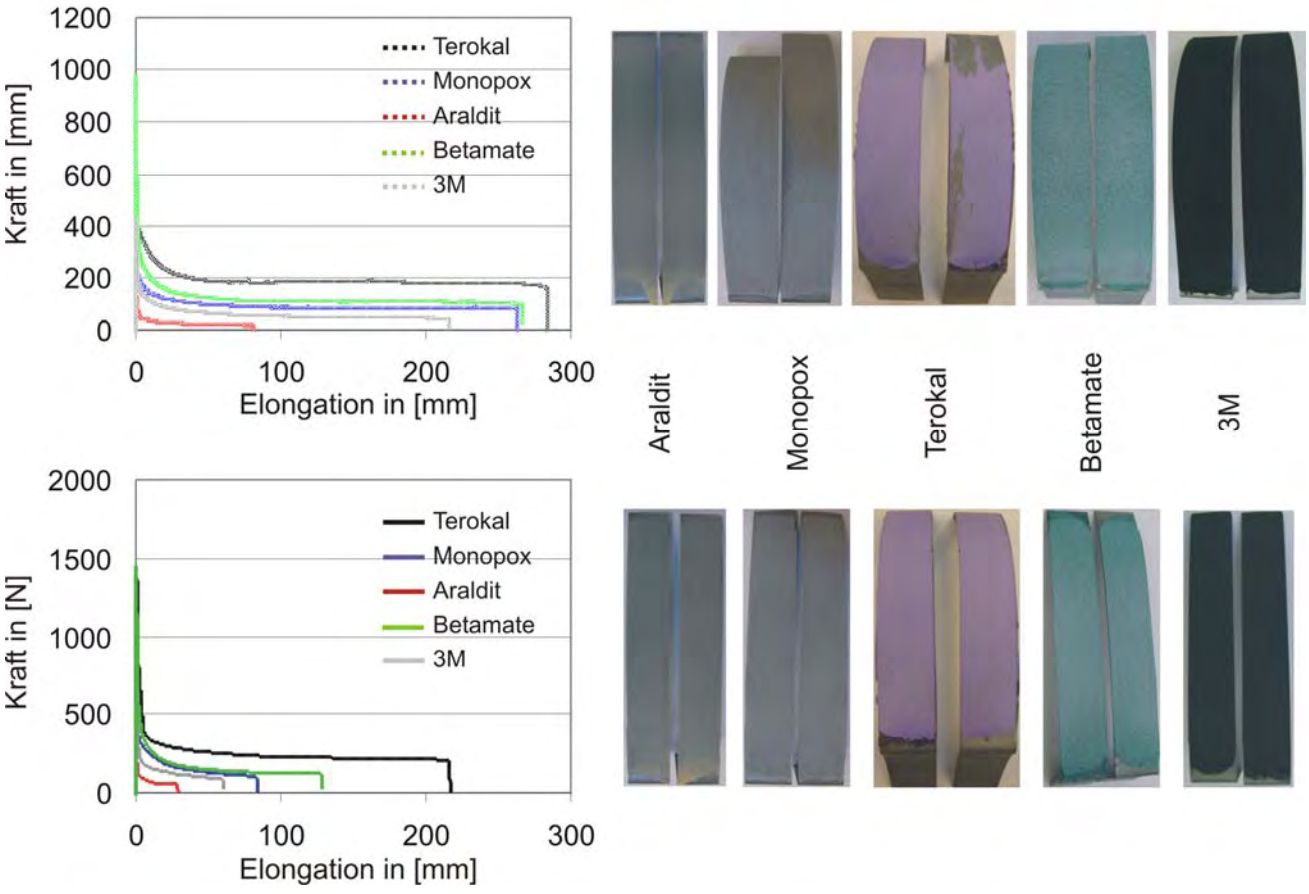


Fig. 52: Peel Test analysis

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Conclusions
The standardized peel and shear tests are good methods for qualifying the adhesives potential. To guarantee a homogenous breaking pattern and minimize the steels deformation influence the tests have to be modified. After the shear and peel testing, the following conclusions can be made:

- The commercial obtainable adhesives are able to develop strong adhesive forces with the galvannealed coating, since all adhesives fail cohesively in the peel test. Monopox and Terokal show the highest potential, reaching the highest stress/strain levels. Hence there is no need to improve the adhesion to the zinc surface by pre-surface treatments in this project.
- Spacers have to be used in the shear test to guarantee a centric load transmission so that the adhesives do not fail prematurely.
- Ductile deformation of the base material causes an undefined stress behaviour which correlates most likely with an adhesive failure between the base material and the galvannealed coating. This presumably is a result of the originating peel forces.
- Backing strips have to be used when dealing with steels reaching their yield stress and creating a complex stress condition.
- The adhesive Betamate and 3M fail prematurely when using the standardized and the modified shear test on both steel materials. Hence this adhesive will not be used in further investigations, since a failure in the GA coating has to be provoked in order to improve the galvannealing process.
- Araldit shows a poor behaviour in the shear and peel test and will not be used in further investigations.
- Monopox and Terokal are reaching the highest stress strain levels in the shear and peel tests.
- The adhesives curing temperature does not effect the shear test results.

Thermal Characterization (DSC analysis)

A DSC analysis has been used to characterize the thermal behaviour of the used adhesives. While heating a cured adhesive up to 200 °C in a 30 minutes time span thermal reactions like crystallization, glass transition, decomposition etc., is measurable by the adhesives enthalpy alteration.
Fig. 53 shows the DSC analysis of the 3M and Araldit adhesives. While both adhesives seem to decompose at temperatures over 200°C the adhesive Araldit has a low glass transition temperature of about 40°C. Furthermore at temperatures of about 160°C some adhesive contents seem to melt or evaporate.
Terokal, Betamate and Monopox, Fig. 54 and Fig. 55, show a similar thermal behavior as 3M with a glass transition temperature of 65°C and a starting decomposition at temperatures slightly over 200°C.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

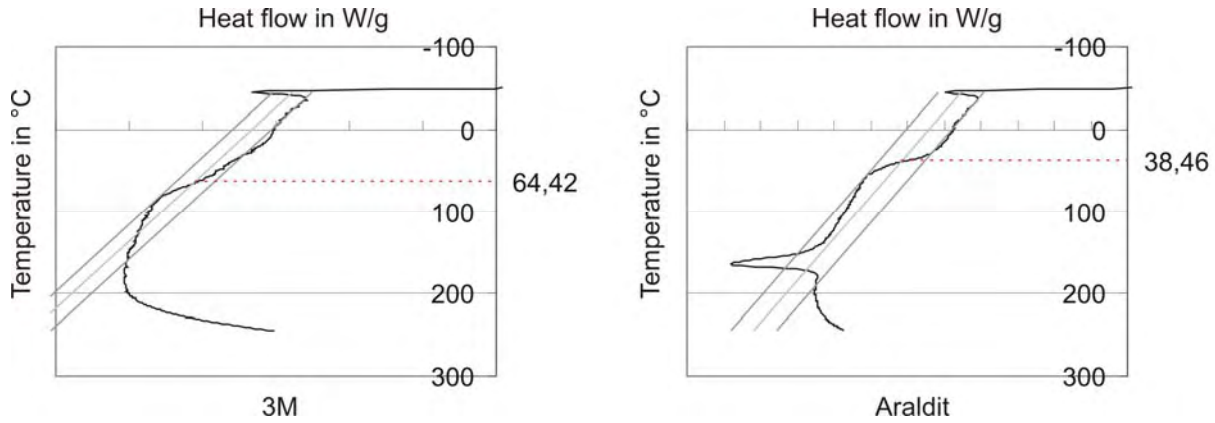


Fig. 53: DSC analysis of 3M and Araldit

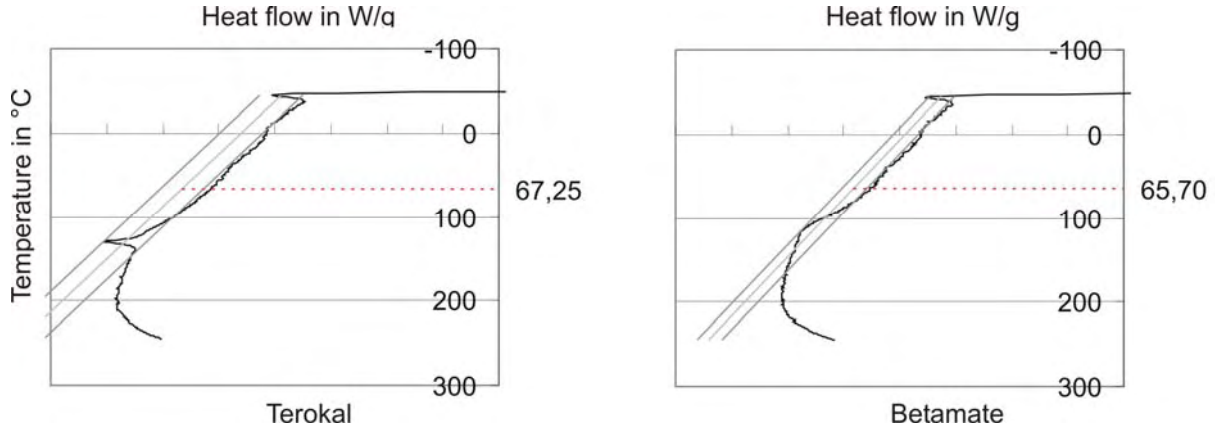


Fig. 54: DSC analysis of Terokal and Betamate

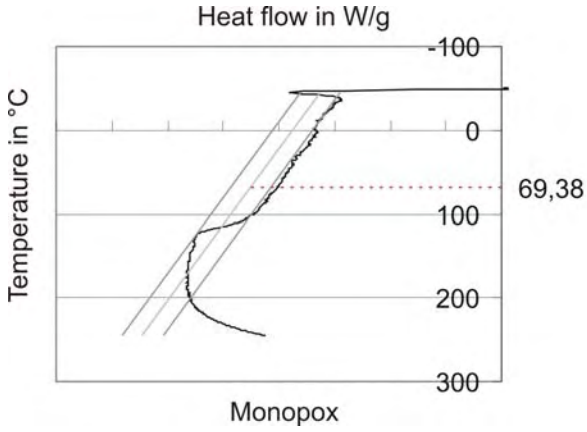


Fig. 55: DSC analysis of Monopox

Rheological characterization

To analyze the dependency of the viscosity in respect of Shear stress and temperature, the adhesives were tested on a Rheometer. The results of the rheological analysis are shown in Fig. 56. The test results of Terokal, Monopox and Betamate show a thixotropic behaviour,

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

while the viscosity of Araldit increases with higher shear rates. The 3M adhesives test results do not show a continuous curve progression.

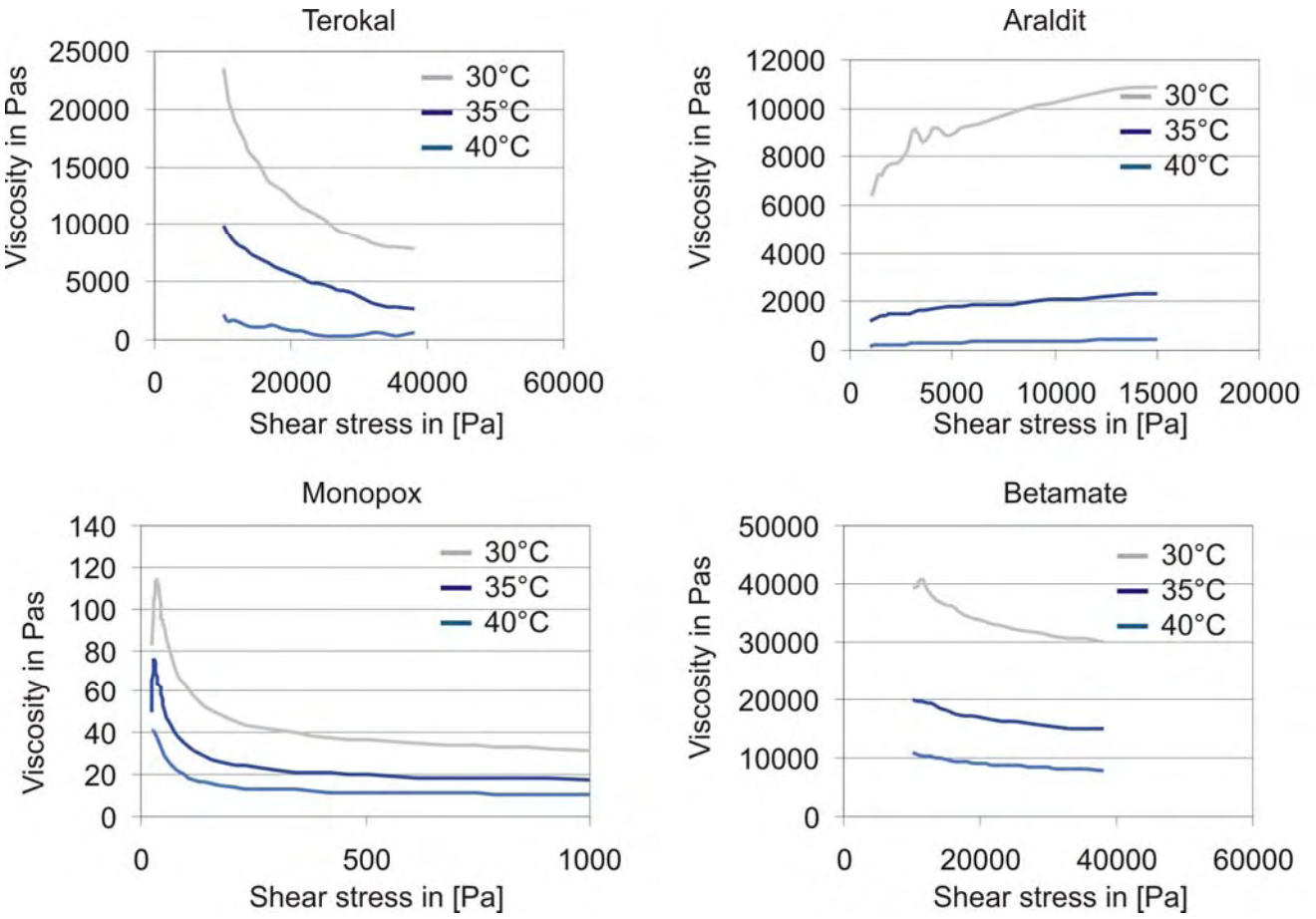


Fig. 56: Rheological test results

Since Araldit and 3M are two component adhesives which start curing immediately after mixing the two components, the test results cannot be proper analyzed since they are altered by the starting curing process.

Conclusions

- Except Araldite the adhesives show a similar and for epoxy resins typical thermal behaviour
- At temperatures about 160°C contents of the Araldite adhesive melt or evaporate so that this adhesive is suboptimal for high temperature applications in comparison to the other analysed adhesives.
- Terokal, Monopox and Betamate show a thixotropic behaviour.
- Rheological results of Araldit and 3M cannot be analyzed since the starting curing process influences the measurements.
- Viscosity of all adhesives decreases when increasing the temperature. Monopox has the smallest Viscosity at room temperature allowing for an application without warming up the adhesive.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Conclusion

- **Task 1: Review of literature on galvanneal coating adherence relative to adhesively bonded peel and shear strengths**

Most literature is dealing with IF steel substrates and only few citations deal with AHSS. The IF steel having the highest potential for lap shear test of adhesively bonded GA steels is a Si-added Ti-Nb IF steel grade. Concerning multiphase AHSS like DP or TRIP, no prediction can be issued so far. Thus, ILZRO sponsors are asked through a survey about the AHSS grades they'd supply for experimental investigations.
It was found that the larger the roughness of steel substrate, the greater the interfacial shear strength is. Thus, investigations on the influence of the substrates roughness will be carried out in the project framework.
Γ/Γ_1 formation seems to be dominant in respect to adhesion behaviour of galvannealed steel sheet. Hence, the experimental investigation of the influence of the nature and amount of the Γ and Γ_1 phases at the substrate/coating interface would be very interesting. This could be a key for improving GA coating adhesion and behaviour during lap shear test.
Investigations should focus on low GA temperatures in order to insure satisfying coating properties like good formability and low powdering.
The Fe composition dependence of the adhesion strength of galvannealed may not be the best way to evaluate the real cause of the improvement in adhesion strength. Better would be to consider the relative phase make up and composition of the coatings instead or additionally to the Fe content dependence of the GA adhesion and shear strengths. Also, when considering coating Fe content, investigations concerning the suitable content, i.e. low or high, to be targeted in order to reach improved strength in accordance with satisfying powdering resistance should be realized.
The most important parameters influencing the galvanneal make up have to be investigated: substrate roughness, GA temperature and exposure time.

- **Task 2: Production of galvannealed samples for adhesive bonding**

The experimental method for processing reliable GA samples at RWTH Aachen is staying an open question, since processing homogenously galvannealed specimens is the most important milestone to investigate adhesive bonding behaviour.

- *Preliminary continuous GA trials*

Only one IR-furnace parameter combination is possible during an experiment with Rhesca HDPS type EU A II at IEHK. Thus, recrystallization annealing and GA-step cannot be simultaneously optimized. But, additionally trials need to be carried out with varying IR-furnace parameters, sample position in IR-heater and sample cooling position. Hopefully a parameter combination ensuring sufficient GA coating quality will be found, otherwise GA must be performed with intermediate solidification of molten Zn.

- *Preliminary GA trials with intermediate Zn solidification*

The inhomogeneous diffusion between the both samples sides is due to the fact that the coating on one side is always slightly thicker than on the other side and/or because of sample bending during Rhesca simulations. Anyway, additional trials will be carried out with pre-load to avoid sample bending effect (also with IF steel).

- *Subtask 2.1: Selection of steel grades for galvannealing*

The experimental study will first begin with an IF steel grade (for continuous GA simulations to investigate the substrate roughness influence) as discussed during the GAP meeting in

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

November 2008 before proceeding with an advanced high strength steel (AHSS) with a multiphase microstructure (DP steel grade would be of great interest for the GAP sponsors). The AHSS grade will depend on GAP sponsors wishes and supplying possibilities, since the literature review didn't point out specific conclusions on AHSS.

- **Task 3: Shear strength testing of adhesively bonded samples**

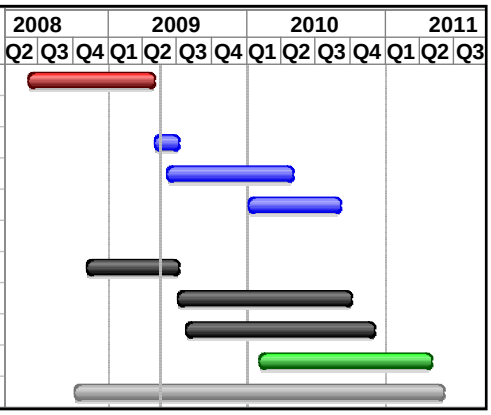
- *Subtask 3.1: Characterization of adhesives*

The adhesives characterization shows that the development of sufficient adhesion forces to the galvannealed surface is not a problem. All used adhesives fail cohesively in the peel test. Furthermore the simple shear test showed, that to analyze the fracture pattern and qualify the compositions resistance to shear stress, spacer have to be used to guarantee a centric load transmission. Otherwise peel forces elevate the developing stress peaks at the adhesive layer edges and result in a premature cohesive failure of the adhesive or a partially delamination of the GA coating.
When dealing with steel sheets which reach their yield stress in the shear test, for example the US600 used in the mechanical characterization, the plastic deformation of the steel initiates a complex stress condition which results in a mixed fracture pattern, which is more difficult to analyze. Thus, backing strips have to be used to minimize this effect. The backed up shear test shows a complete failure in the zinc coating with the Araldit, Monopox and Terokal adhesive while Betamate and 3M fail prematurely, so that a characterization of the GA coating is not possible. Consequently Betamate and 3M will not be used in further investigations.
To identify the fracture pattern failure location in the zinc coating an analysis tool is needed which is on the one hand able to differentiate the different phases within the GA coating in a sufficient resolution and on the other hand allows for a quantification of the whole fracture pattern.
EDX scans have been carried out to try to differentiate the different phases according to their iron respectively zinc content. These investigations show that although it is possible to quantify the iron and zinc content, the results have to be treated very carefully since it is possible that the electron beam of the EDX simulates deeper located layers which alter the test results. Consequently an alternative solution has to be considered.
The thermal and rheological analysis showed only minor variations between the different adhesives.
After considering the whole adhesive characterization the adhesives Monopox and Terokal have been chosen to use in the remaining project. While the Betamate and the 3M adhesive did fail prematurely when using the backed up shear test, Araldit has handling disadvantages because of its two components. Furthermore compared to the other used adhesives, Araldit shows a poor peel behaviour.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224

Program for next period

Work program	*State*
Task 1: Review of literature	Finished
Task 2: Production of galvannealed samples	
Subtask 2.1: Selection of steel grades	Ongoing
Subtask 2.2: Galvannealing of samples	
Subtask 2.3: Focus on a limited number of GA	
Task 3: Shear strength testing	
Subtask 3.1: Characterization of adhesives	Near to completion
Subtask 3.2: Production of adhesively bonded samples	
Subtask 3.3: Shear testing of samples	
Task 4: Microstructural and chemical analysis	Preliminary investigations
Task 5: Analysis and reporting	Ongoing



Subtask 2.1: Selection of steel grades for galvannealing

- Sponsor supply of IF steel (voestalpine)
- Integration of AHSS in the work program by survey concerning GAP sponsors requests and comments

Subtask 2.2: Galvannealing of samples

- GA simulations with intermediate liquid Zn solidification with industrial “GI” IF steel (like with Al% in Zn-bath as Al% used for GA) using Trebel with pre-load to avoid sample bending effect
- Additional continuous GA trials with Rhesca (varying IR parameters, sample position in IR-heater and sample cooling position)

Task 3: Shear Strength testing

- Production of adhesively bonded samples with the provided IF-steel from voestalpine. The backed up sample preparation will be used
- Shear strength testing of the bonded specimen

Task 4: Microstructural and chemical analysis of high quality samples

- Alternative fracture pattern analysis tools will be checked in order to find alternative to EDX
- Student research project to this subject will start in November 2009

Acknowledgment

Thanks to Mr. Strutzenberger (voestalpine) for his comments and coaching. Thanks also to ThyssenKrupp Steel (Mr. Norden) and US Steel (Mr. McCosby) for material supply.

www.iehk.rwth-aachen.de
info@iehk.rwth-aachen.de
Univ. Prof. Dr.-Ing. Wolfgang Bleck
Fon +49 241 80 95783
Fax +49 241 80 92224