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ADHESION OF ZINC HOT-DIP COATINGS

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Abstract

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The work is focused on verification of quality adhesion of zinc coating. It describes elements which affect quality and adhesive solidity within the coating. For assessment itself it will be neccessary to get know the basic elements which can affect adhesion of hot-dip coating which will be essential for choosing suitable samples for verification itself. These elements characterise acoustic responses during delamination coating. They affect elements influencing progress of signal. In research there is also a summary of existing methods for testing adhesion of coatings. As a result a new proposal of a new method comes out for purpose of quality testing of adhesion zinc hot-dip coating. The results of verification of this method are put to scientific analysis and findings lead to assessment of proposed method and its application in technical practise.
The goal of this contribution is also include to proposed methodology testing adhesion zinc coating by nondestructive diagnostic method of acoustic emission (AE), which would monitor characterise progress of coating delamination of hot-dip zinc from basic material in way to adhesion tests would be practicable in situ. It can be enabled by analysis and assessment of results acquired by method AE and its application within verification of new method of adhesion anti-corrosive zinc coating.

Keywords: hot-dip galvanizing, adhesion, adhesion testing methods, acoustic emission

INTRODUCTION

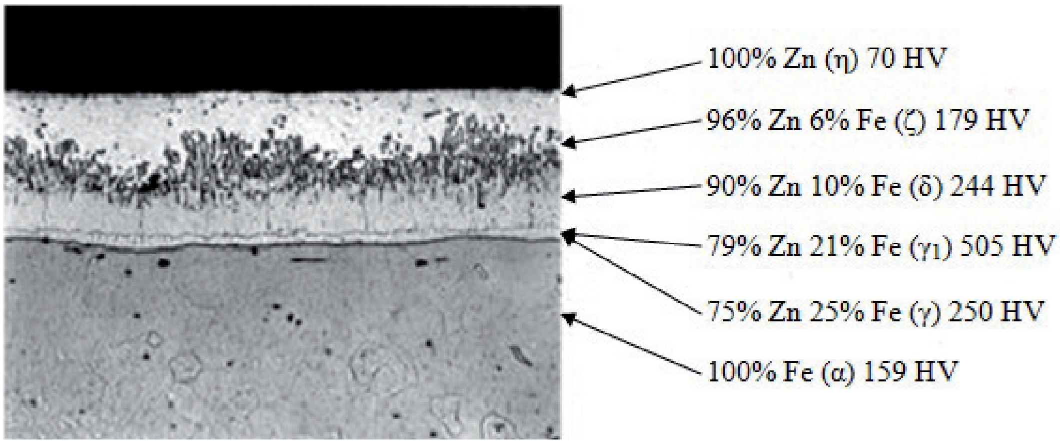
Zinc coating, hot-dipped applied, is often used preventive protection against corrosion. The reason of using is not only the price or life but mainly technological and technical elements. It includes especially ease of process of application and strong preventive resistance even in case of coating damage. Nowdays modern technologies enable huge diversity in way of application. The dimensions of products are not limited either. The important aspect of protection is coating adhesion which is difficultly measurable. From physical side it can be said the adhesion is a complex of mechanical powers related to square unit. The coating is bound to surface of constructive material by them. From chemical side, the adhesion can be defined as a intermolecular bond, both chemical and physical power on point of contact surfaces in unevenness and material pores (kmm.cz, 2013; adheze.cz, 2013).

It is evident that if we talk about adhesion, we mean the bond power, both mechanical and physical, between coating and material surface. In this case any endeavour to determine the size of surface adhesion should lead to measuring of intensity of this bond.

The hot-dip galvanizing is one of the most frequent way of application zinc coating on steel surface. It uses both in continuous processes in production and aslo in piece production.

The layer of zinc coating is typical of relatively evenly thickness, good adhesion and also estetic appearance. The disadvantage is a neccesity to keep technological nature at galvanized part, size of parts (with respect to dimension of vessel with zinc bath) and necessity to make a surface adjustments in zinc plant (Roubíček, 2003; Horák, 2009).

Application of hot-dipped galvanized material is related to anti-corrosion resistance even in cases when compactness of coating is broken. In view of the fact of formability of zinc layer the sheets with coating are suitable for deep drawing (ksp.tul.cz, 2013). The significant feature is creating stronger layer of coating on the edges of parts than on the straight surface. Here the hot-dipped coating distinctivly differ from paint coating (Eriksson, 2005).



1: *Intermetallical phase ordering in anticorrosion zinc coating*

Intermetallic phase Fe-Zn

In reaction of molten zinc with steel appears Fe-Zn on boundary so called intermetallical phases which differ by quantity of contained zinc in phase and crystallographic structure. The distribution of intermetallic phases (Fig. 1) has a fundamental influence on final feature of coating (Průšek, 1985; Horák, 2009).

Mechanical analysis of coatings system – base is hard. The detailed recognition of mechanical phases features, its bonds and intermediary stages features is insufficient untill these days (sciencedirect.com, 2012).

Influence of coating adhesion

Adhesion of coating is basic requirement. Without firm fastening coating on material surface the correct function is disabled (Průšek, 1985).

Surface adjustment before galvanizing

The preparation of material surface is demonstrated after certain time when the coating compactness is damaged (Kreibich, 2007; Průšek, 1985). The removal of organic substances (oils,fats) then soil and corrosive products and roughening surface according to requirements other technology lead to improvement of conditions, quality and quality galvanizing (Bartoníček, 1980; Kreibich, 2007). With the help of mechanical adjustments the corrosive products and other dirt is removed. These are the main things which cause bad adhesion of zinc coating (Kreibich, 2007; Průšek, 1985; Kreibich, 1996). It can be assumed that for coatings adhesion the depression has a greater significance than protrusion of profile. These enable better fastening of coating. The protrusions has a negative significance because the coating is reduced in its place. Here it comes to damage (Sedláček, 2011). The thickness of basic material has influence on thinckness of excluded coating which can affect adhesion of basic material itself (Křemen, 2010). Another important process of metal surface adjustment before hot-dip galvanizing is dipping. Inappropriate chemical cleaning has a straight effect on adhesion (Černý, 2001).

Chemical composition influence

At deoxidized steel the crystals of outer layer create space among crystals which are in form of small grain or long columnar crystals. The zinc can penetrate to steel surface without problem and that is way the reaction in zinc bath can run quickly for all time dipping and created zinc coating is without cavities or pores at the same time. Silicon in combination with phosporus causes in certain concentrations reactivity increasing of iron with zinc. It comes to creation of grown through zinc – alloy Fe-Zn and creation of coating at thickness exceed minimal values set by norm of ČSN EN ISO 1461. Besides darky matted shade coating is also characteristical by relatively big brittleness. The brittleness of zinc coating grows even with increasing admixture of Sn in zinc bath (Horák, 2009; Wiegel gruppe, 2003).

The stell deoxidized by Al is a steel which has a collective content of silicon and phosporus under 0,04%. There is a reaction at this steel only between zinc and iron where there is a alloy layer Fe-Zn-Al. The coating remains relatively thin (Stryž 2013). Alloying elements in steel (Mg,Al etc) can oxidize before process of zinc coating application. The surface covered by oxides worsen its wettability in liquid alloy bath which can lead to points flaw. Coating adhesion to steel and ability resistance against corrosion is decreased. It has a cardinal impact at the moment of creating of galvanized piece (sciencedirect.com, 2012). Carbon causes that during higher concentration the reaction Fe-Zn increases together with thickness of coating. By this fact the adhesion of coating can be affected. During higher content of sulphur (over 2%) is reaction of zinc and iron so quick that all phases cannot be created and that is way zinc coating is not used for these steels (Křemen, 2010). Alloying elements are often added even in zinc liquid alloy for reason of improving certain features of coating. Aluminium creates thin film Al_2O_3 on the surface of liquid alloy.

This prevents quick oxidation with air. It helps decrease thickness of excluded coating at silicum steel. Aluminium also increases shine of coating and resistance during bending. Inhibitory layer is created at the same time and reaction between Fe and Zn is reduced. In case the Al source is unsufficient (for example Al is lesser than 0.10%), inhibitory layer will be thin and more Fe will go to melted zinc and creates compounds (surface layers) Fe-Zn. If we use tin as a alloying element in zinc bath we can achieve significant lowering of coating thickness at constructive steel with content of Si 0.034 till 0.30% (Sciencedirect.com, 2012; Konstrukce.cz, 2010).

Thickness of zinc coating is affected by the time of galvanizing. The hot-dipping is usually between 1.5–5 minutes, at the great parts the hot-dipping can reach up to 10 minutes. The continuous galvanizing is exception because in this case the hot-dipping is very short. The hot-dipping has an influence on reaction Fe-Zn and also on thickness of coating but only at silicon and deoxidized steel – in this case the thickness of coating depends on hot-dipping. If the time of hot-dipping by silicon is exceeded at deoxidized steel the ideal thickness of coating is not reached. The main sign of this coating will be brittleness (Vítek, 2010; Horák, 2009). Absorbed hydrogen (during dipping) is very reactive. In case of infiltrating into basic material the brittleness is increased. Removing hydrogen is realized either by thermal adjustment (180–200 degrees for two hours) with double galvanization and chroming or in the way that galvanization runs into final thickness of layer with following tempering on 210–240 degrees for 1–2 hours. (there is a excretion of intermetallic phase y1 from faze y followed by discontinuity in construction of atomic bars. The phase is very hard with minimum of plasticity which cause flaking of coating (Ruml, 1981; Horák, 2009; Jansen 2002).

Straining

Presence of mechanical tension in material causes corrosive crackling. That is a process running at effect of static tractional tension and corrosive processes. It causes degradation of basic material and violates adhesion of coating to the basic material. The galvanization carries out after processing of form (Bartoníček, 1966). The hot-dip coating is not suitable at parts which are exposed to temperatures over 250 degrees at heating lasting more than one hour. It applies especially in cases when these parts are mechanicaly stressed. It was found out that in these cases there is a flaking of zinc coating on boundary of intermetallic phases o and y (Horák, 2010).

After creating of zinc coating (at temperature about 419 degrees) there is a cooling both coating and heated steel which is covered with zinc up to room temperature. That is what this different expansivity of zinc and steel is a source of tension on their boundaries. The greater part of this tension is shifted to shape of plastic deformation of zinc grains, the rest makes microcleft on boundary of grains. Mechanical tension of part can cause widening of microcleft created on boudnary of grains. If there are greater parts FeZn in zinc coating there is a creation of clefts in zinc coating which spread along parts. We can eliminate this element by having AL content in bath in concentration from 0.15–0.20% weight, which during process creates inhibitory layer. This layer slows down diffusion of iron to phase n and its transformation to strong phase layer (Sciencedirect.com, 2013).

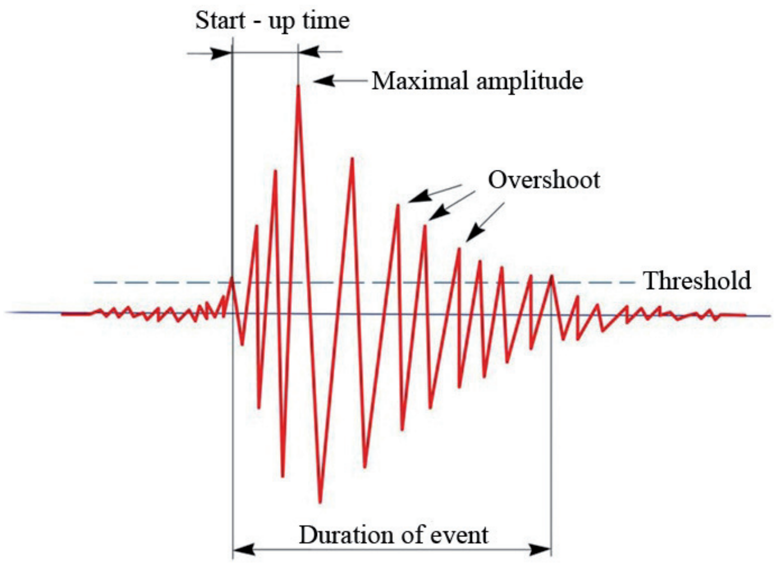
Acoustic emission

Acoustic emission is a physical element when in solid particles appear inner elastic waves created by dynamic process. The elastic waves are recorded by sensors placed on surface of body. AE is a significant in field of NDT because it can detect transformation inside of material and enables warning before collaps. The one of the most common case of appearing sudden breakdown in running is a damage of material homogenity. AE can detect micro damage inside of material during operational conditions. To get a realible results there is a necessity to pay attention to choice and quality of fastening and calibration of sensors. In other case it can happen that it will not be able to distinct anything between humming and real signal of acoustic emission.

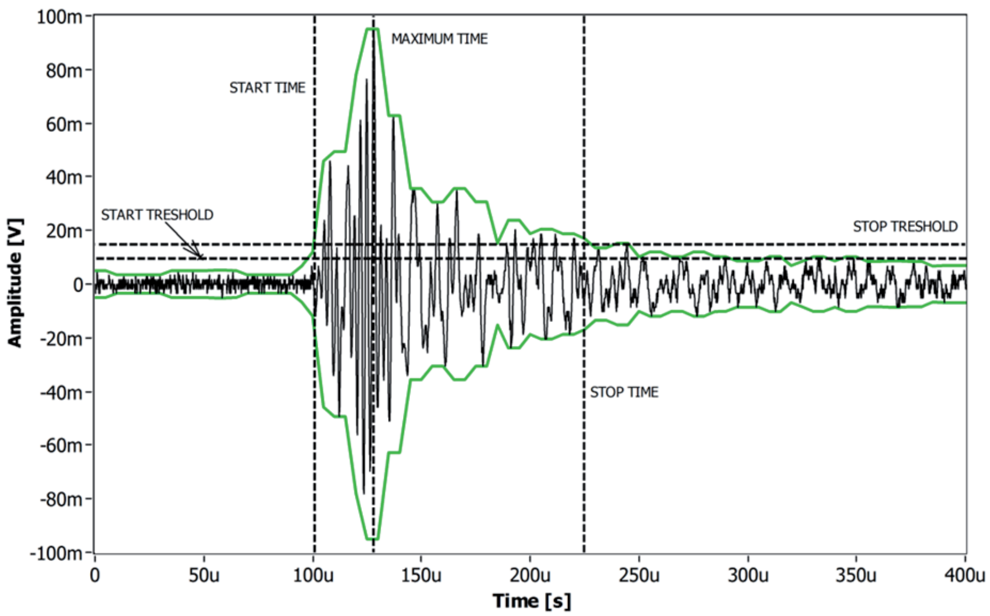
The method AE detects the progress of process and its characterization. It works with „listening“ of acoustic activity. The limitation of acoustic emission method consist in unability of detection static defects. It is related the necessity to apply the incentive to reach acoustic reaction. The activity AE is usually growing with followed straining (Kaiser's effect). This straining causes the appearance of new defects which are permanent. That is why the AE test is not repeatable. The element called „event“ causes rise of acoustic emission (Fig. 2). It is for example dynamical appearence of microcleft, shif of dislocation etc. On the other hand „source“ of acoustic emission is a physical origing of event. It can be a progress of defects, phase transformation, dislocation shift, inner friction, formation and spreading of clefts. The released energy is transformed on mechanical impuls which is spread in material in form of elastic voltage wave.

One isolated hit of electrical signal of acoustic emission is caused by detection of shock wave of one event. According the feature there is a distinction: continuos signal does not plummet below set threshold value and uncontinuous signal which has a feature of time-separated packages. The uncontinuous signal of acoustic emission is created by time isolated hit order which last in between nanoseconds and miliseconds. The spectrum of impuls in frequence is very wide. The signal is a type of muffled vibrations which have quite big amplitude. The typical sours of uncontinuous signal at acoustic emission is changing cleft in material.

On the recorded signal AE is capitalizing so called acoustic interference of frequence units kHz up to



2: *The example of acoustic event of signal*



3: *The example of acoustic event of signal*

hundreds of kHz. The intensity of this interference descends with growing frequence (Vlašic, 2011; Dvořák, 2010; Vlček, 2009; Preditest.cz, 2013). During processing AE the signal is first separated from initial and final noise part of record. The amplitude (Fig. 3) of analytical signal is calculated and it is cover of original signal and it enables by oscillation to remove the study of the cover (Cietec.eu, 2013; Chlada, 2008).

The standart assesment of AE is done according to the norms ASTM E596-91, which according to the accoustit activity, acoustic intensity and power seriousness classify sources of acoustic emission into there levels of seriousness.

A. Insignificant (passive) source AE is such a source when signs fit the presence of insignificant defect.
B. Potencial significant (active) source AE is source when signs fit the presence of initial defect which is significant from the point of view of future spreading due to real straining parametres.

Critical significant (critical active) source AE is source whose signs fit the defect development.

The development of breach carries out in local weak parts of material like welds, pasted jolts, segregations, inclusion etc. – nonhomogenic places. Nevertheless in case that material seems to be homogenic, the meassurements AE shows on its homogenity when weak parts in material are presented in form of bubbles, clefts, inclusions of filth or corrosive parts (Přibáň, 2010).

Testing adhesion of zinc coatings

It regards to set the complex of forces related to square unit which binds the coating to the surface. The adhesion is able to reach the high level closing to solidity of the coated or basic material (Průšek, 1985).

The norms to test the adhesion set the testing methods rather by ductility than its adhesion. Furthermore these methods only show the results like – suitable vs unsuitable absent quantifiable results. The objective testing method for quantifiable assesment of adhesive power at zinc coating misses. There are several methods proposed. The common atribut is destructive test which means permament damage of testet coating or material itself. Yet the parametr of adhesion is related to bond power between coating and material. This bond can be meassured only by separating of coating! (Havránková, 2005; Sciencedirect, 2012; Katrenjak, 2011).

ČSN EN ISO 1461:2009

The appropriate adhesion with metallized component is characteristic for hot-dip galvanizing. The component should be able to hold the manipulation which relates to thickness of coating and normal use of product without creating plates or flaking. This norm sets that the test is supposed to be arranged between contractor of coating and customer (ČSN 1461, 2010).

ASTM 633-69 (ČSN EN 582)

The purpose is to set the solidity of coating or jolt solidity of sprayed coating and undercoat metal. It is based on sticking to counterpart of same shape. The whole set is put to axial and tensial straining afterwords. It is similar at classic test of tensile (Houtková, 2003; ČSN 582, 1995).

According to Ambroz

The main thing is pulling out of pin at average of 20mm from content which contains desk and pin. Before spraying of coating there is a mutual connection. The whole preparation is refilled of device whose purpose is to set the numeral level of adhesion which uses the principle of flexible deformation of dynamometric element (Houtková, 2003).

According to ASTM A 123

To set adhesion of zinc coating there is a neccessary to use cutting or nick using knife blade. The adhesion should be considered as unsuitable in case the coating layer is flaked showing basic material (ASTM A123, 2012).

Bending

It is an assesment of coatin appearance of five successive bends at 180 degrees at jerk about 8mm average. During flaking of coating the adhesion is assesed as unsatisfactory. The cleft formation is not significant (Průšek, 1985).

According to Ollard

The coating at thickness of 2.5 mm is transfered onto point. The end of the stick is machined on lathe in such a way the coating sprayed on point is supposed to overlap. The gauge is a power which is neccessary to rip annullus of sprayed coating from surface of testing stick. The power is checked on tearing machine in axile direction (Průšek, 1985).

The methods of heat impact

It is a difference of coefficient of linear expansivity of basic material and metallic coating. For coatings Zn-Fe is set temperature 180 degrees. After that, the samples will dip into water of temperature 20 degrees. The formation of blebs or flaking coating is monitored (Průšek, 1985).

EXPERIMENTAL PART

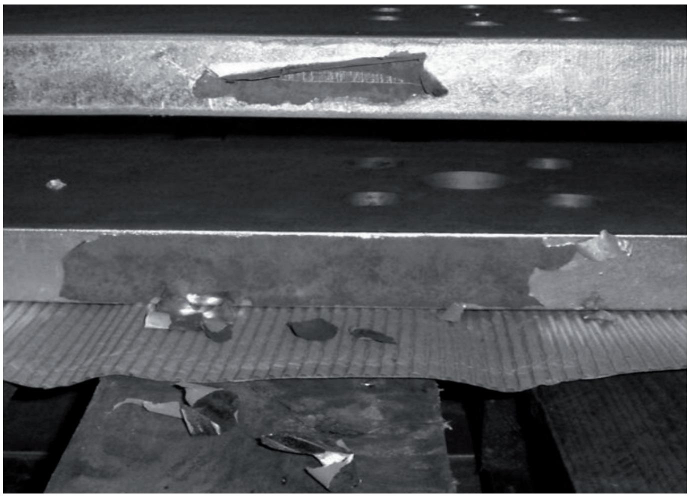
The goal is to propose and verify the original method of quality testing of adhesive solidity hot-dip galvanized coatings. This testing method should have defined conditions comparable for this type of tests. It means that identical results should be achived assuming the defined steps are followed.

The state is simulated which causes on common conditions spontaneous dehohesion of coating (Fig. 4). After disrubtion the coating gradually flakes from surface of basic material and it bends outward the material.

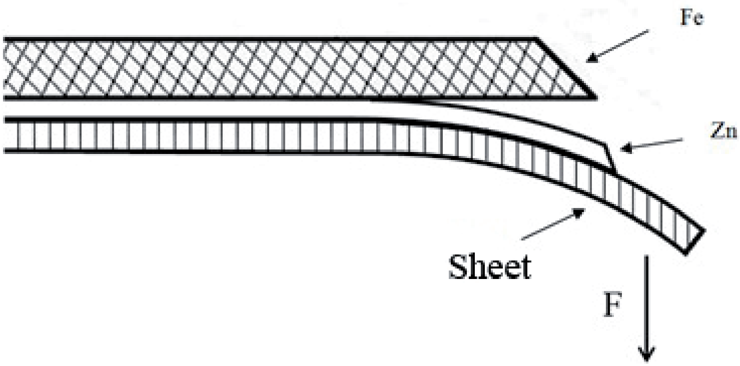
Primarily it is inevitable to solve the way of fastening the coating. At the same time it is neccessary to define adjustments of coating for test adhesion by uniaxial strain. The fastening of sample is to enable safe sticking of acoustic emission sonds which will record waves spreading in proggres of delamination.

The solving is to stick coating to thin metal which should gradually separate from the base at tensial load and it would flake layer of zinc coating at the same time. The presumption is that used adhesive can play the role of adhesive phase (Fig. 5).

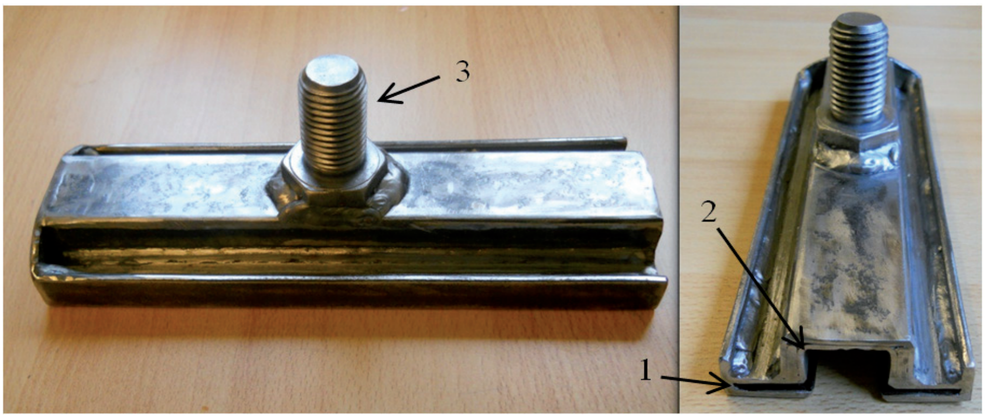
The choice of adhesion is basic fact at the proposed method. The used adhesion has to have higher adhesive and cohesive solidity than supposed anchor adhesiveness of coating on steel sheet. The adhesion Hysol 9514 has been chosen for its tensile strenght, which is 44 MPa up to thickness 3mm (does not have to be guaranteed minimal level of adhesion layer affected for exampple by uneveness of adhesived spaces).



4: *The example of real delamination of coating*



5: *Delamination scheme of zinc coating after sticking on steel surface*



6: *The preparation for meassurement by tractional adhesive strenght*

Testing preparation and sample

The construction of preparation for fastening of tested sample is pictured on the Fig. 6.

The opening is made for insertion of sample; thin sticked sheet is set to lower part of preparation. The raised part 2 has a purpose of cavity to place a sond of acoustic emission. The bolt M30 (3) has a purpose of joint locking to the preparation of meassuring tensiometric top. The flaking is solved by trackling of wire at the thickness of 3mm with ball seating.

The hot-dipped galvanized sheet from deoxidized AL steel has been used for production of testing sample. The Pb and Sn have been used as a dipping elements in zinc bath. For the function of „flaked“ was chosed the steel from AL steel at the thickness of 1 mm. The surfaces was roughened, degreased, washed and dried. The edge of galvanized stell was filed on belt sander at the angle of 45 (modelling of flaking coating). Sticked samples were strained by

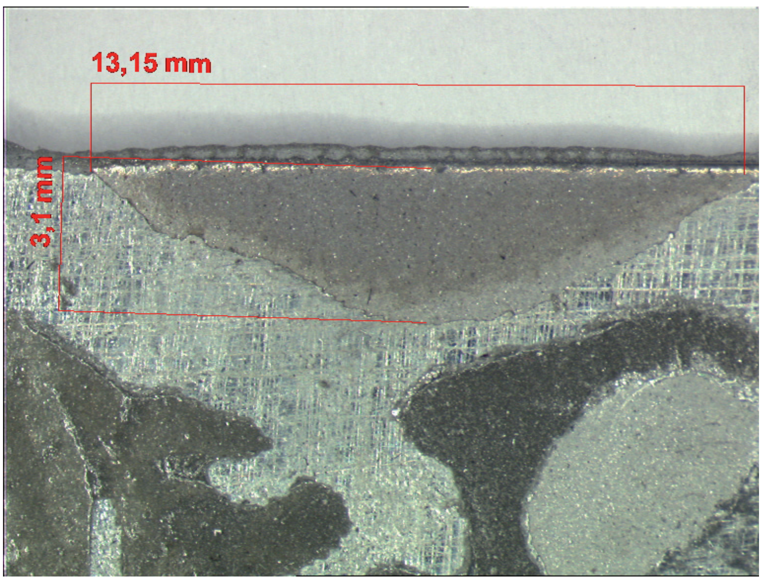
7: *Tractional sample ballasting, detail of sheet fastening*

weight at 1kg because of displacing air bubbles). The samples were strained like that for 24 hours.

After hardening of adhesion in furnace were the samples let to eliminate additional voltage. The coating was cut in. The are of zinc coating determined for verfication of adhesion remained compact just towards to the base and steel sheet. The verfication of method was done on 36 samples in ÚFM AVČR in Brno (three series). The sonds of acoustic emission were placed on tested sample from the opposide site along the sheet sticking. After that the sample was inserted into preparation and fastened to device crosspiece by means of wire. The result of fastening is on Fig. 7.

RESULTS AND DISCUSSION

The majority of samples (when the breech of adhesive joint occured at high level of adhesive coating solidity!). The breach of sticked layer of cohesive-adhesive character. The examples of macro-delamination of Zn coating at samples which are visuably assesable are on the Figs. 8, 9. The flaking is recognizable only at three samples just in form of pitting. – on some places sticked places at the size of on tenth milimeter (observalbe under binocular). The samples with large delimination of coating were chosen for assesment of strenght progress and acoustic emission. Firstly, the typical measurring conditions were described then stated



8: *Primary and secondary delaminaton of sample no 2 from first measurement* characteristical photos of delamination (coating or adhesion), graphically analyzed strenght progress of delamination with time and finally analyzed progrres of acoustic emission.

Sample No. 2 (first measurement):

The speed of crosspiece shift 50 mm. The flaking of zinc coating occured in two places. Adhesie delamination of zinc coating and base tranfered in breech of adhesion of adhesive-cohesive character. The breech of adhesion spread out whole tested place with exception of other lesser flaking of zinc coating (Fig. 8).

The trend of strenght surge was growing up to level ca. 300N. After that the decrease followed which kept between 50N and 100N. In view of the fact the locality occurence of flaked zinc is at the beginning of sample we can make a conclusion that power which is necessary to delaminate hot-dipped coating was at level of 300N (Fig. 9).

Interval of surge at unstuck zinc coating reflects the interval of two amplitudes of acoustic signal. Two initial amplitudes of the most powerful energy of acoustic signal (RMS – blue line) correspond with two locations of flaked zinc. The amplitudes present the response of delamination of coating. The third biggest energy of acoustic signal between 60th and 70th second is caused probably by the change of character breech of pasted joint (Fig. 10).

The increase of numbers of acoustic events is obvious as well. It reflects the progress of cumulative frequency of events (evn – black line). The number of occurence of events and energy of acoustic signal in bigger at initiation of deflection and spreading microclefts on interfacion of zinc coating. The occurence and spreading microclefts within pasted joint shows lower energy of signal and gradual increase of events frequency.

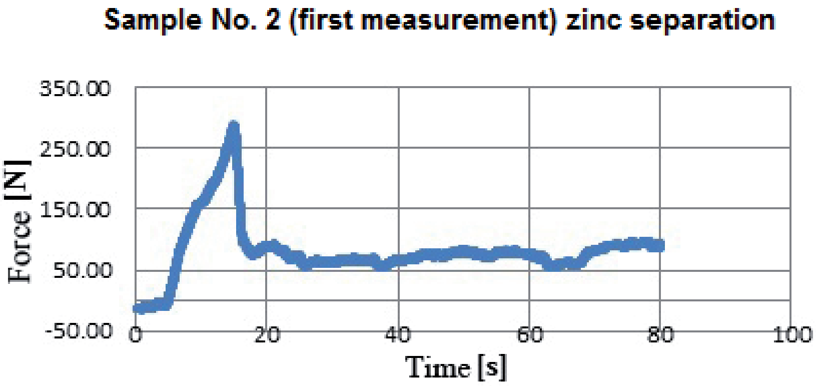
The sample No. 2 (second measurement) – little decohesion

The speed of crosspiece was increased up to 100mm.min. The flaking of zinc coating occured only in one place. (ca. 2.3 mm, Fig. 11). The lost of adhesion was mainly caused by means of adhesive mechanism.

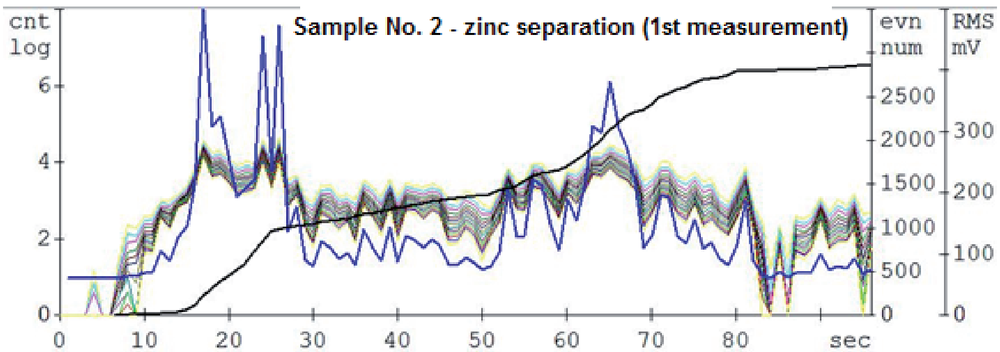
Action of force at destruction of pasted joint (flaking of zinc coating) reached up to 250N (Fig. 12).

During comparison of force increase with progess of acoustic emission (Fig. 13) we can observe congruence of both progresses. The force increase at delamination of coating response to amplitude of energy of acoustic signal (RMS) and cumulative frequence of events (evn) is also higher. We can observe the confirmation of Kaiser effect from the frequency of events and progress of force. (during decrease there is no surge of emission parametres)

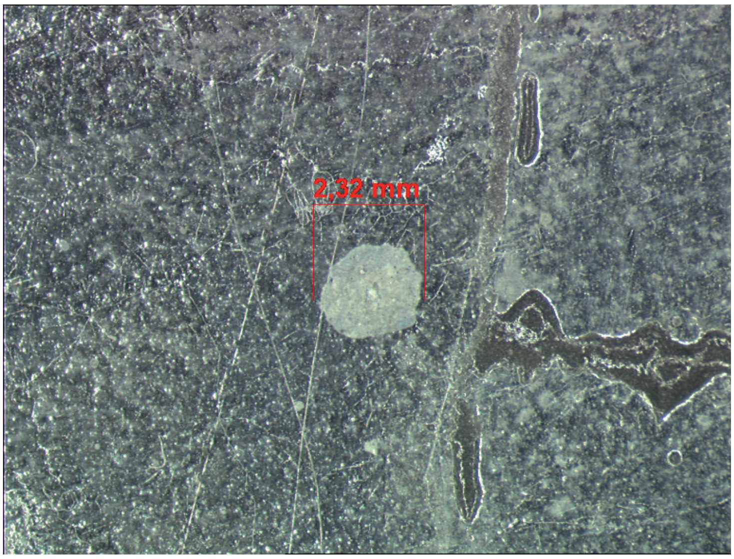
The record of action of force confirms that delamination (Fig. 14). Zn-coating occurs over level of 250N. If the levels of transmitted force at phase between sheet and zinc coatig (adhesion solidity) is higher than 250N, we can overstep the boundary of



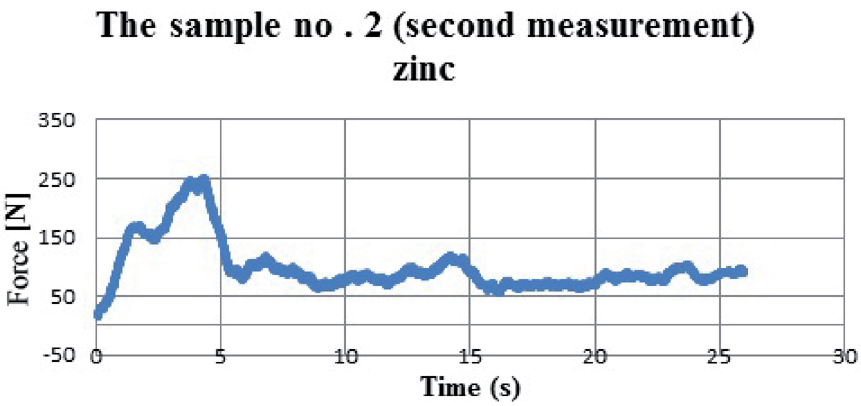
9: *Characteristic progress of power at delamination of coating.*



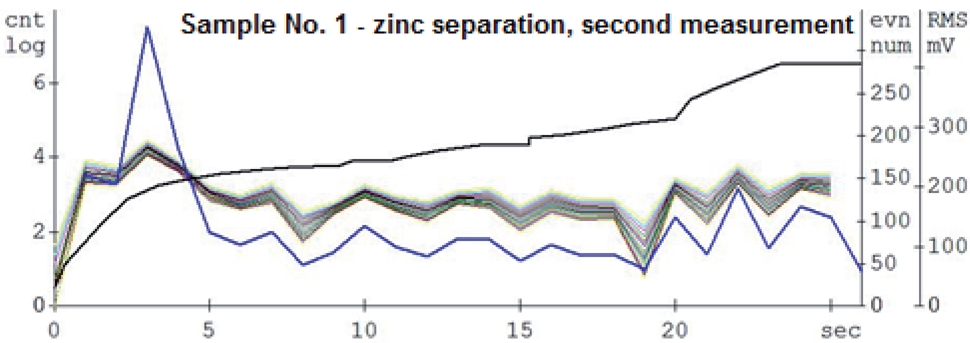
10: *Record AE during decohesion of sample no 2 from first measurement*



11: *The separation of zinc coating of sample No. 1 from second measurement*



12: *Action of force response of sample No. 1 from second measurement*

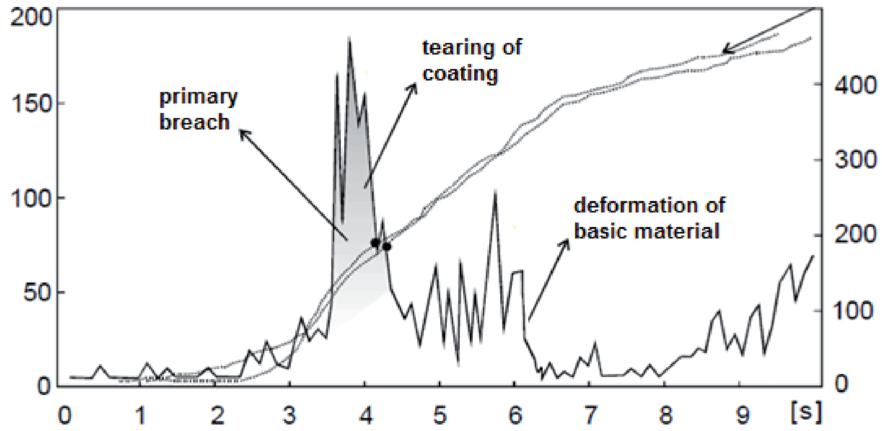


13: *Acoustic response during delamination of sample No. 1 from second measurement*

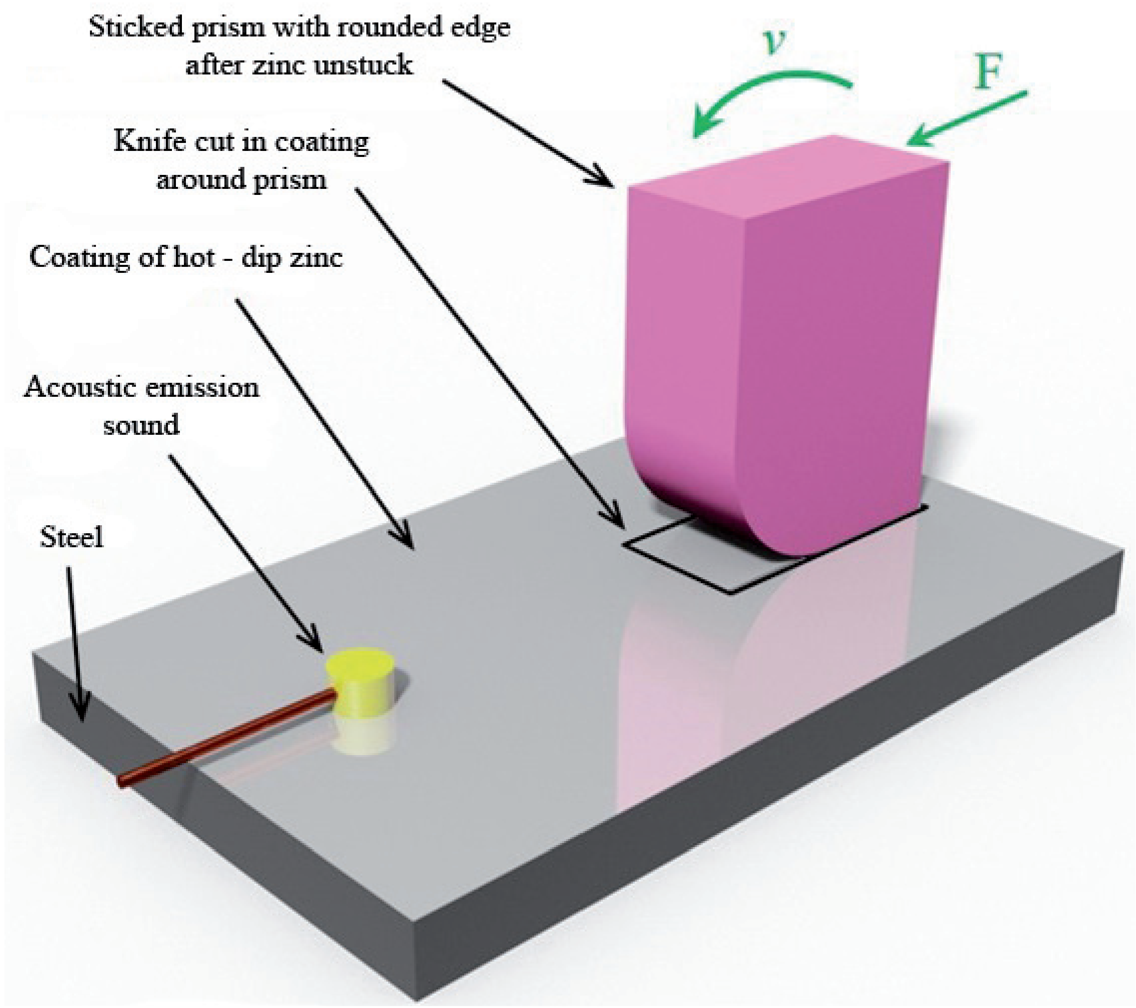
anchor force of zinc coating and basic material. In this case there is a only delamination of hot-dipped zinc coating.

Little flakes of coating in form of pitting appeared at samples reaching force over 180N. We can assume that these little flakes occured in places of unsufficiant anchoring of coating to steel surface. It could be caused for example by presence of filth on the edge of hot-dip coating and basic material. The lowered adhesion of zinc coating could be caused even by uneveness of steel sheet before galvanizing. The presence of microprotrusion (flash) can launch local increase of coating. It probably affects even the coating adhesiveness.

In cases the force did not reach more than 170N we can assume there were inappropriate conditions



14: *Record analysis AE at delamination*



15: *The proposal of device for measurement „in situ"*

of solidity at the sides of adhesion. It can be expected that the solidity of pasted joint should come out from possibility of adhesion which should be able to transmit action of force at least about 250N at proposed testing configuration. From the results of acoustic emission is obvious that the biggest energy of acoustic signal was caused by delaminate zinc coating. While the cumulative frequency of occurence of acoustic events depended even on formation and way of spreading of cleft in pasted joint.

CONCLUSSIONS

The unstandart process of pasting and toughness of sticked sheet for delamination seem to be problematic. When it comes to loading which is followed by realising before hardening, the bending of sheet occurs due to elastic deformation. It can be elaminated by using thinner sheet (for example 0.5 mm). There is also possibility to harden adhesive in furnace under loading. This process counts on using furnace which would be able to hold the samples even with preparation.

The transmitted way of method can by applicated even for research of adhesive solidity in laboratory but also in terrain for example quality verification of hot-dip galvanized coating for bridge or any other constructions. Modification of method for terrain measurement consist of record strenght absence. The flaking of zinc coating is assessed on the base of visual and from record of acoustic emission. The static bend of sticked prism which is bended around rounded edge can be used to breech the adhesion (Fig. 15). The research study of this modification is necessary for choosing adhesion without any hardening! From the AL record we can assess adhesion breach as a significant surge of energy of acoustic signal.

For verification of results from terrain would be suitable to create static study of loaded galvanized coatings with use of AE.

SUMMARY

The literary research was made in this work. It deals with both structure, features of individual phase of hot-dip zinc coating and factors which can affect quality and adhesive solidity of these phases. The sources of acoustic response were described in material togehter with factors having influence on progress of signal.

The goal was to propose method of testing the quality of adhesion of zinc coating on hot-dip galvanized coating verify the suitability in laboratory. The verification was made on standard tractional device with help of preparation and commercial sondc of acoustic emissions which was specially constructed for these measurements. The lack was proven in the field of pasting technology. The proposals were set in results disscusion which should suppress these lack.

The verification clarified that proposed method has a potential for valuable results gained by combination of levels measurement of adhesive solidity and analysis of acoustic emission signal. It is obvious the used hot-dip galvanized coatings has a feature of good quality of adhesive solidity. The solidity was intensified by presence of aluminium (AL deoxidized steel) which created in reaction with iron and zinc strong phase Fe-Zn-Al (only roughly verified SEM). Furthemore the elements Pb and Sn were used to decrease wettability in zinc bath which lowered thickness of excluded coating and increased its adhesion to steel. These factors ensures higher adhesiveness of coating which is fastened on the steel surface with help of simple diffusion process within structure of intermetallic Fe-Zn phases.

The proposed method for modification is sutiable even for testing of coatings in terrain and it could be used in large use related to mapping of actual quality adhesion of galvanized coatings.

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