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Research Article

THE VISUAL METHOD IN QUALITY ASSESSMENT OF SINGLE-LAP ADHESIVE JOINTS

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ABSTRACT

This article presents selected criteria for the quality assessment of single-lap adhesive joints formed on 0.66±0.04 thick hot dip zinc coated sheets with the hot dip zinc coating thickness equal to 18 μm. Three types of epoxy adhesives and three variants of the mass of adhesives were tested (1 g, 2 g and 3 g). The work intended to assess the capacity of the visual method with regard to evaluating joint quality, allowing for the presence and the size of spew fillet, joint dimensions and the effect of spew fillet on adhesive joint strength. The strength of analysed adhesive joints was determined in destructive tests.

Keywords: adhesive joints, visual method, spew fillet, adhesive joint strength

INTRODUCTION

Adhesive joints control process comprises a number of operations during which factors regarding joint production and finished joints are scrutinized [1-6]. Due to the fact that adhesive bonding is regarded as a special joining process it requires specific approach, accounting for the necessity to apply a variety of different test methods to control the finished joint, as well as joint formation processes [7-11]. That is why a complex control of adhesively bonded joints is most frequently required, and this may include the following tests [12-16, 17, 18]:

- dimensional and shape accuracy evaluation (with naked eye or with the aid of appropriate equipment),
- adhesive layer control (defect detection in the adhesive layer itself),
- various strength tests,
- operation tests.

The visual method is one of the non-destructive testing methods (NDT) and is regarded as a fundamental scientific method [1, 7, 19]. Visual testing is defined as a process of observation conducted with naked eye (direct method) or aided (indirect method). The process aims at verification of conformity of the test object with specifications described in, e.g. norms, commissioning requirements and other regulations. Commonly assisted by other non-destructive testing methods, it constitutes the first basic control stage, consisting in visual inspection for discrepancies observable with a naked eye or with the aid of appropriate tools, usually optic, frequently preceded by the measurement of their characteristic dimensions [1, 7, 11]. Visual inspection requires compliance with basic conditions regarding the correctness of control, such as: having required qualifications, good eyesight, appropriate luminous intensity (500 lux) as well as the ability to distinguish and interpret inaccuracies [20].

Non-destructive testing methods do not cause the failure of or defects in a joint or adherends. There exist a number of NDT methods [9, 13, 14, 19]: visual, penetration, ultrasonic, eddy current, radiographic or magnetic particle testing methods. Nevertheless, not all the methods are suitable and can be applied to adhesive joint inspection. Particular non-destructive testing methods are characterised in numerous papers.

This article undertakes the analysis of the visual method, applied to inspect single lap adhesive joints. Single lap joints are widely applied in a wide range of branches of industry, therefore are a frequent subject of research [3, 16, 21-24].

EVALUATION CRITERIA IN THE VISUAL METHOD

The analysed criteria include:
a) adhesive layer quality,
b) the presence of spew fillet,
c) joint dimensions,
d) the shape of formed joints.

Adhesive joints were subjected to inspection with regard to each of the aforementioned criteria, taking into consideration their characteristic indices. It was decided that adhesive layer quality would be evaluated with regard to: gas bubbles, adhesive layer discontinuity, delamination, colour and consistence.

The spew fillet was analysed in terms of its size and position. The literature [12] reports a large amount of studies into spew fillet shape with regard to adhesive joint strength, for instance, R.D. Adams and J.A. Harris presented the effect of local geometry changes at the edges of the overlap in single-lap joint and LDR Grant et al. [24] studied various shapes of spew in single-lap and T-joints.

Joint dimensions evaluation comprised the following measurements: overlap length, adhesive layer thickness, joint length and width, as well as perpendicular and parallel orientation of adhered elements [1, 11, 24]. L.D.R. Grant *et al.* [24] examined the behaviour of adhesive joints when various parameters, such as overlap length, adhesive thickness and the geometry of the edge of the overlap region were studied. Finally, Belingardi *et al.* [25] and Andreassi *et al.* [26] studied various aspects of adhesive fillet.

Factors influencing the dimensional and shape accuracy of adhesive joints, which in turn contributes to adhesive joint strength, include [1, 2, 27, 28]:

- the type of joint (shape and dimensions of the joint and adhered elements),
- surface preparation of adherends (method, treatment parameters),
- the type of adhesive applied (the number of components and such properties as: form, chemical composition, viscosity, cure time, curing agent);
- curing conditions (temperature, pressure, time, equipment applied, method of applying pressure).

The importance of recognising and determining the aforementioned factors, particularly with regard to adhesive joint strength, is highlighted by numerous studies conducted on the subject [27-31]. Shenoy *et al.* [22], who characterised fatigue damage in single-lap adhesive joints subjected to constant amplitude fatigue loading similarly used the spew fillet. Apalak and Engin [21] determined the effect of the adhesive fillet size around the adhesive free-ends on damage zone initiation and propagation in aluminium single-lap and double-lap joints in experimental and numerical test. Wu *et al.* [28] presented a set of differential equations for the analysis of joint-edge loads in dissimilar adherends with different thicknesses and lengths. Wang *et al.* [29] developed a method based on the successive boundary stress concentration in adherends of adhesively bonded joint with square edges or spew fillets at the ends of the overlap. Magalhães *et al.* [23] presented the results which showed important stress variation along the adhesive thickness near the overlap edge. Rudawska [16] investigated the influence of the adherends thickness on the lap length of aluminium alloy sheets bonded joints.

TEST METHODOLOGY

Adhesive joint characteristics

The tests were conducted on 0.66±0.04 mm thick shear loaded single-lap adhesive joints of hot-dip zinc coated sheet (DX51D+Z275MA [32]), with coating thickness of approx. 20 μm. The test sample, together with its dimensions, is presented in Figure 1.

The dimensions of the analysed adhesive joint were the following: sheet length ls = 100 mm, sheet width 20 mm, sheet thickness ts = 0.66

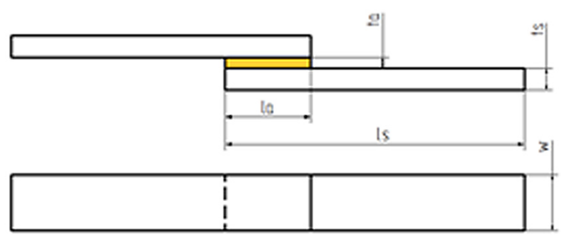

Fig. 1. Tested adhesive joint

mm, calculated overlap length lo = 17 mm. Adhesive joints measurement results obtained following curing process are presented in the section Test results.

Joint formation conditions

The initial stage of the experiment consisted in the preparation of the surface for subsequent adhesive joining. The surface treatment operation of degreasing was performed with Loctite 7036 degreasing agent and consisted in triple application of the degreasing agent. Each procedure was followed by the removal of excess of the agent together with contaminants, whereas after the last application it was allowed to evaporate for approx. 3 minutes until the surfaces dried off.

Subsequently, the adhesive was applied on one of the adhered surfaces, immediately after it has dried off, and further assembly performed. The laboratory conditions for joint forming were as follows:

- temperature: 21± 2°C,
- humidity: 22± 2%,
- applied pressure: 0.03 MPa.

The adhesive joint was then cured under pressure of 0.03 MPa for 72 h. Joint seasoning time was equal to 144 h, in identical conditions as in joint formation.

Adhesives characteristics

The tests were conducted on three adhesives in three adhesive composition mass variants, of 1 g, 2 g and 3g, nevertheless, the three adhesive composition mass variants selection was dictated by the volume of joint surface area. The mass of adhesive was determined with ONYX OX-120 analytical balance with an accuracy of 0.0001 g.

Loctite 9466 is an industrial epoxy adhesive characterised by medium viscosity. It offers considerable strength and extended work life. Once mixed, this two-component epoxy cures at room temperature to form an off-white adhesive layer characterised by high peel and sheer strength. Fully cured provides high resistance to a majority of chemicals and solvents and, simultaneously, electrical insulation [33]. Superb strength properties of this epoxy adhesive designate it as a great solution for bonding various polymers and metals in industrial applications. Moreover, its long work life enables introducing adjustments to the alignment of assembled elements.

Loctite 9484 is an industrial epoxy adhesive of medium viscosity. Characterised by high strength and medium work life once mixed it cures at room temperature forming flexible vibration and shock resistant grey adhesive layer. Fully cured provides resistance to a majority of chemicals and solvents and, simultaneously, electrical insulation. Applied predominantly in polymer, metal, glass, wood, ceramic and rubber material bonding, this adhesive is suitable for joints where flexibility is required. It is applicable for joining heterogeneous materials operating in low static stress and high dynamic stress conditions [34].

Loctite 3430 is a two-component epoxy adhesive which cures promptly at room temperature. This general purpose adhesive produces high strength on a number of adherends. Due

Table 1. Selected properties of uncured material [33-35]

Properties	Adhesives		
	Loctite 9466	Loctite 9484	Loctite 3430
Chemical type (resin)	epoxy	epoxy	epoxy
Specific gravity ≅ 25 °C	1.00	1.30	1.14- 1.20
Viscosity, Brookfield - RVT, 25 °C, mPa·s (cP):	15,000-50,000 (spindle 6, speed 20 rpm)	60,000-175,000 (spindle 7, speed 10 rpm)	18,000-28,000 (spindle 5, speed 2.5 rpm)
Chemical type (hardener)	amine	polyamide	epoxy
Mix Ratio, by volume - resin: hardener	2:1	1:1	1:1
Mix Ratio, by weight - resin: hardener	100:50	100:85	100:100
Pot life, 22 °C, 100 g, min	60	40	4

Table 2. Selected properties of applied adhesive (cured material) [33-35]

Properties	Adhesives		
	Loctite 9466	Loctite 9484	Loctite 3430
Tensile Strength, ISO 527-3, N/mm²	32	15	36
Tensile Modulus, ISO 527-3, N/mm²	1,718	161	3,210
Shore Hardness, ISO 868, Durometer D	60	55	70
Elongation, ISO 527-3, %	3	50	2

Note: Data collated in Table 2 regard properties of adhesive cured for 7 days at 22 °C.

to its good gap filling properties it is perfect for rough or loosely fitting metal, ceramic, rigid plastics or wood [35].

Adhesive joints of zinc coated sheets were formed with tree types of two-component epoxy adhesives: Loctite 9466, Loctite 9484 and Loctite 3430, the selected properties of which are presented in Table 1 (for uncured material) and Table 2 (for cured material) [33-35].

Loctite 9466 and 9484 adhesives require 24 hours in room temperature in order to produce high strength. The process, however, can be accelerated by applying higher temperature. The increase of shear strength in the function of time and temperature for the adhesives is presented in Figure 2 [33-35]. Hysol® 3430™ is a two component, clear epoxy adhesive which cures rapidly at room temperature after mixing.

Adhesive compositions tested in experimental study contain an identical chemical resin compound - epoxy, yet differ in terms of the curing agent chemical type, which results in their different properties. Different curing agent requires different adhesive components mixing ratio by volume and by weight. Another factor taken into particular consideration was the working time of the adhesive, which necessitated performing joints within a couple of minutes after mixing the components of adhesives. Moreover, attention was given to the amount of adhesive mixed, due to the fact that excessive amounts of adhesive and ambient temperature above 22°C contribute to shortening the working time, owing to, *inter alia*, emitting excessive amounts of heat.

Adhesives used in the tests are delivered in dual packages (cartridges), facilitating dosing and preparation. Mixing and dosing was performed with special dispensing device – a double syringe and a static mixer. After mixing the adhesive was applied on one of the adhered surfaces.

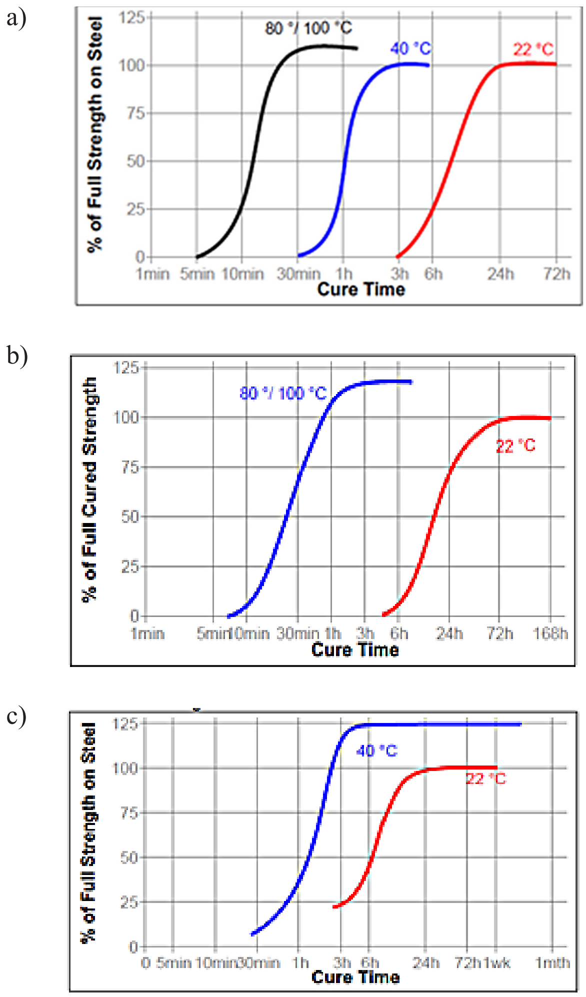

Fig. 2. Grit blasted structural steel adhesive joints strength (ISO 4587) formed with: a) Loctite 9466, b) Loctite 9484, c) Loctite 3430 [33-35]

Measuring instruments applied in the visual method

The visual inspection was conducted with the following instruments:

- Sylvac System digital calliper with the resolution of 0.01mm, measurement range of 0-150 mm, and of 0.03 mm accuracy;
- Sylvac System digital calliper with the resolution of 0.01mm, measurement range of 0-200

mm, and of 0.03 mm accuracy;
- WG-9 Automatic WELD Skew-T Fillet Weld Gauge,
- a magnifying glass,
- inspection mirrors,
- a stereoscopic microscope.

The visual method evaluation criteria

The evaluation of dimensional and shape accuracy with the visual method was conducted as per detailed evaluation criteria collected in Table 3.

In the tests the evaluation of spew fillet presence and its dimensions was conducted. Moreover, the control of dimensional and shape accuracy along with adhesive layer thickness repeatability was performed. The analysis of adhesive layer formation accuracy included such factors as: gas bubbles at the edge of adhesive layer, adhesive layer discontinuity, delamination, colour and consistence. The visual method applied in these tests does not enable the observation of adhesive layer discontinuity, delamination or gas bubbles at the edge or along the length of the overlap. Since the aforementioned defects in the adhesive layer cannot be detected with the method in question, other NDT methods should be applied. The visual method, however, proves useful in evaluation of the colour of the adhesive layer, which provides initial indication of the correctness of cure (in general, of the correct temperature of the process), due to the fact that a well-cured adhesive is of characteristic colour. If too dark, the colour could indicate exceeded suggested cure temperature, similarly, too light may be a result of lower than recommended temperature. The evaluation of adhesive layer consistence, which depends largely on proper conduction of cure process, is yet another important element of the visual inspection. Brittle or excessively hard adhesive layer may be indicative of overcuring, whereas overly soft adhesive – undercuring.

The presence of spew fillet

Some researchers [2, 12, 21] point at a positive effect of spew fillet at the edge of the overlap. It has been found to increase joint strength as a result of reducing boundary stress concentration. Therefore, it is advisable to leave spew fillet, on condition that structural considerations allow for its presence.

Dimensions and shape of adhesive joint

Dimensional and shape accuracy inspection of formed single-lap adhesive joints comprised the following elements: overlap length, adhesive layer thickness and other dimensions of a joint, such as length and width. In addition, the perpendicular and parallel orientation of adhered elements was evaluated.

TEST RESULTS AND DISCUSSION

Visual inspection of the spew fillet presence

Tests of spew fillet were divided into two stages. The first stage involved determining the presence of spew fillet as well as measuring its

Table 3. Dimensional and shape accuracy criteria for adhesive joints

Dimensional and shape accuracy criterion			
1.	Adhesive layer quality	1.1	Discontinuity
		1.2	Delamination
		1.3	Gas bubbles
		1.4	Colour
		1.5	Consistence
2.	Spew fillet	2.1	Size
		2.2	Location
3.	Adhesive joint dimensions	3.1	Overlap length
		3.2	Adhesive layer thickness
		3.3	Joint length
		3.4	Joint width
		3.5	Perpendicular orientation of joint elements
		3.6	Parallel orientation of joint elements
4.	Adhesive joint shape	4.1	Strain
		4.2	Undulation of the surface of adhered elements
		4.3	Clamp marks on the surface of adhered elements

area using the visual method. Secondly, the colour and consistence of spew fillet were evaluated depending on the type of adhesive.

The presence of spew fillet at the edge of adhesive layer is presented in Figure 3. After curing process the spew fillet was not removed, on the contrary, it was left with the assumption that structural considerations allow for its presence.

The presence of spew fillet was visually evaluated. It was observed that the presence as well as size of spew fillet are contingent on both the mass and the type of the adhesive used for adhesive joint formation.

The influence of adhesive on spew fillet presence and size

The visual method applied in evaluating the presence of spew fillet indicates that fillet size depends on the type of adhesive. Test results regarding variability of spew fillet surface area depending on adhesive mass variant applied are presented in Figure 4.

The analysis of test results presented in Figure 4 allows to observe that for the adhesive composition mass variant of 1 g the presence of spew fillet was noted only in the case of Loctite 3430 and not in the other two adhesives, Loctite 9466 and 9484. Considering the 2 g variant comparable results of spew fillet surface area were observed for all adhesive compositions. Most dissimilar results of fillet surface area measurements were noted in the case of 3 g of adhesive. Moreover, this variant produced highest spew fillet surface area which was, in addition, notably two and three times higher in the case of Loctite 3430, 113.74 mm^2, as compared to Loctite 9466 and Loctite 9484 respectively.

In conclusion, it was observed that together with the increase of the mass of adhesive composition increases the surface area of spew fillet, whose size, *nota bene*, depends on the type of adhesive. In the case of Loctite 3430, the viscosity of which is the lowest, spew fillet was present in each of the adhesive composition mass variants. Simultaneously, Loctite 9484, the least viscous composition, produced spew fillet of the smallest surface area in 2 g and 3 g variants of the three analysed adhesives and mass variants. The results were obtained in identical curing conditions in terms of pressure applied while curing. It therefore appears that the criteria for selection of adhesive should, in addition to the type, include its viscosity with a view to obtaining spew fillet.

Colour and consistence of adhesive layer

In the second stage of the analysis with the visual method, colour and consistence of adhesive layer were investigated. This evaluation was performed following adhesive joints seasoning time prior to strength tests.

No discontinuity or delamination in the adhesive layer or gas bubbles at the edges of the overlap was observed in the tested adhesive joints. Colour and consistence of the adhesive layer is typical of the type of applied adhesive; once cured it can be slightly transparent to milky white. A magnifying glass, inspection mirrors, lighting and a stereoscopic microscope were applied in the inspection. In conclusion, the joints were assessed as correctly formed, since no adhesive layer discontinuity, inclusions or cracks were noted.

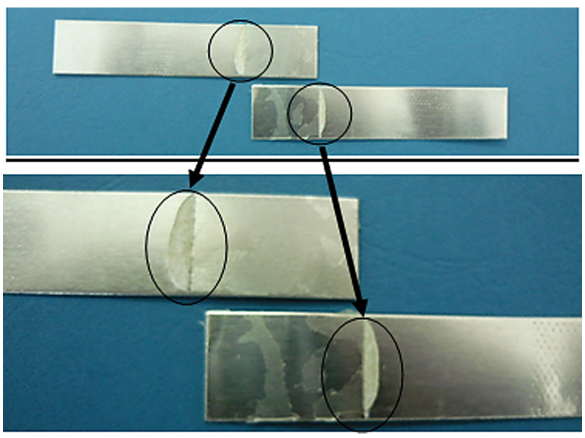

Fig. 3. Spew fillet at the edge of adhesive layer

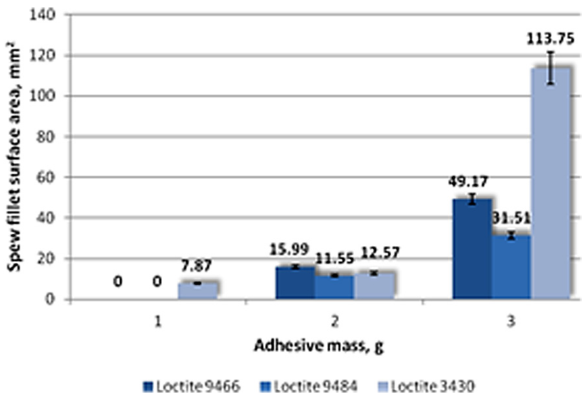

Fig. 4. Spew fillet surface area depending on the mass variant and the type of adhesive

Shape and dimensions of adhesive joints

The following dimensions were measured during the analysis of adhesive joints dimensions: overlap length and width of the adherends, thickness of adherends as well as thickness of adhesive layer. Applied to evaluate the dimensional and shape accuracy through both the measurement of geometric dimensions of the joints as well as shape analysis, the visual method allowed formulating the following conclusions:

1) appropriate accuracy of adhesive joint shape was achieved,
2) overlap length dimensions were within the range of (Fig. 5):
   - 17.40±0.92 mm and 17.93±0.30 mm for Loctite 9466;
   - 16.49±0.47 mm and 17.64±0.38 mm for Loctite 9484;
   - 16.95±0.24 mm and 17.62±0.20 mm for Loctite 3430;
3) different adhesive layer thickness was obtained depending on the type of adhesive and adhesive composition mass variant (Table 4).

The range of overlap length dimensions (Fig. 5), for assumed 17 mm length, may be accepted as a sufficiently accurate dimension. It must be highlighted that joint strength calculations were conducted for actual joint dimensions obtained following curing.

The presented comparison of adhesive layer thickness for different mass variants and types of adhesive (Fig. 6) allows to note that when 2 g of adhesive composition was applied, in each of the analysed adhesives, similar results of adhesive layer thickness measurements were obtained, whereas considerable discrepancies were noted in other mass variants.

For 1 g and 3 g of adhesive the highest thickness was produced by Loctite 3430, which is the least viscous. The lowest differences in adhesive layer thickness were observed for Loctite 9466, characterised by relatively highest viscosity. It might be possible that the higher viscosity the more stable and uniform adhesive layer thickness following curing, as compared to low viscosity adhesives.

Table 4. Adhesive layer thickness measurement results

Adhesive mass, g	Adhesive					
	Loctite 9466		Loctite 9484		Loctite 3430	
	gk, mm	σ, mm	gk, mm	σ, mm	gk, mm	σ, mm
1	0.14	±0.02	0.16	±003	0.26	±003
2	0.16	±0.01	0.10	±0.04	0.13	±0.04
3	0.21	±0.02	0.35	±0.08	0.39	±0.03

gk – adhesive layer thickness, σ – standard deviation.

The effect of spew fillet and its size on adhesive joint strength

Following seasoning adhesive joints were subjected to destructive tests on Zwick/Roell Z150 testing machine. The correlation between the mass of adhesive and adhesive joint strength is presented in Figure 7.

Strength test results indicate that joints formed using Loctite 9466 (Fig. 7) exhibit higher shear strength with increasing adhesive mass. In the three adhesive mass variants, 1 g, 2 g and 3 g, the value of strength equalled respectively 10.67 MPa; 13.28 MPa; 14.10 MPa. The strength of adhesive joints formed with 1 g of the adhesive amounts to 80% of the strength of adhesive joints formed with 2 g of the adhesive, and only 76% of those formed with 3 g of the adhesive. The strength of adhesive joints formed with 2 g of the adhesive is lower by 6% than the strength of joints formed using 3 g of Loctite 9466.

The analysis of test results presented in Fig. 7 leads also to the observation that shear strength

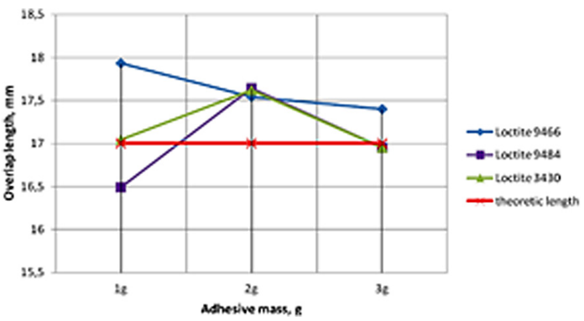

Fig. 5. Overlap length dimensions of theoretic and real adhesive joints

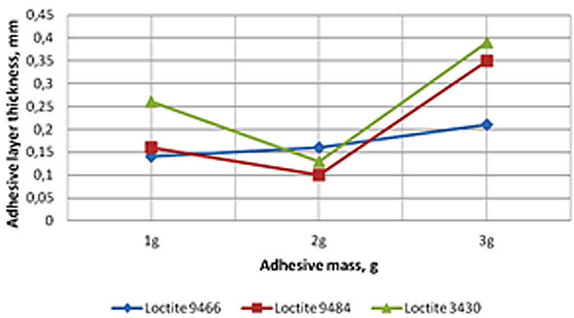

Fig. 6. Correlation between adhesive layer thickness, adhesive mass variant and the type of adhesive

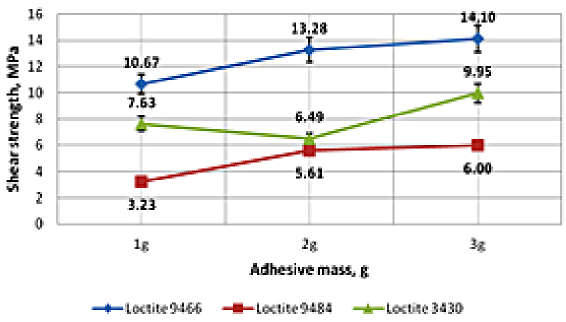

Fig. 7. Correlation between adhesive joint strength and adhesive mass variant

of hot-dip zinc coated sheet adhesive joints increases with increasing Loctite 9484 mass. A considerable difference in tensile-shear strength values between 1 g of adhesive composition mass and 2 and 3 g of adhesive composition mass were observed. The strength of adhesive joints formed with 1 g of the adhesive amounts to 58% of the strength of adhesive joints formed with 2 g of the adhesive, whereas the difference in tensile-shear strength between joints formed with 2 g and 3 g of adhesive composition mass was rather insignificant (approximately 7%).

The presented test results (Fig. 7) prove that in the case of Loctite 3430 highest adhesive joint strength values are produced by 3 g of the adhesive composition, and lowest strength values are obtained for 2 g of the adhesive. The strength of adhesive joints formed using 2 g of adhesive composition amounts to 65% of the strength of adhesive joints formed with 3 g adhesive mass variant, and 85% of strength of joints formed using 1 g adhesive mass variant.

The results indicate that this is Loctite 9466 which produces joints of highest strength. In the three adhesive composition mass variants, 1 g, 2 g and 3 g, the value of strength ranged from 10.67 MPa to 14.10 MPa, whereas, joints of lowest strength were formed with Loctite 9484. Figure 7 (and Fig. 4) display a gradual increase of adhesive joint strength together with the rise of amount of adhesive, which, as mentioned, contributes to the presence of spew fillet and the increase of joint surface area. For both Loctite 9466 and Loctite 9484, in the case where no spew fillet is observed (1 g mass variant), the resulting joint strength was the lowest. The highest strength in the case of all analysed adhesives was obtained when the spew fillet surface area was the highest (3 g mass variant). The results appear to substantiate claims regarding a positive effect of spew fillet on adhesive joint strength.

Furthermore, such a conclusion seems to be confirmed by the comparison of the present test results with published research [2, 12, 21]. R.D. Adams and J.A. Harris [12] noticed that the local edge geometry of the joint has an important bearing on joint strength and that by suitable modification on a relatively small scale, significant increase in joint strength may be achieved. L.D.R. Grand *et al*. [24] proved that a 45° spew fillet creates stronger joint in tension that joint with a square end, especially as the bondline thickness increases. At the thinnest bondline of 0.1 mm, there is very little difference in the strengths of the joint with and without a fillet, nevertheless in the case of a bondline thickness of 3 mm the strength of the square-ended joint is less than half of that with a 45° fillet. M.K. Apalak and A. Engine [21] showed *inter alia* that as the adhesive fillet size was increased the applied force necessary for the damage zone initiation was increased. Accordingly, an adhesive fillet length covering the adherent free edge is advantageous for the joint strength.

CONCLUSIONS

Inspection of adhesive joints with the visual method is among the basic methods of non-destructive testing, therefore, with the assistance of appropriate measuring equipment it should be applied at the very beginning in order to evaluate joint quality. Based on test results of the visual inspection of adhesive joints and destructive test results, the following conclusions can be formulated:

1. The presence and size of spew fillet are determined by the type of adhesive, however, this is the increase of the mass of adhesive composition which results in the increase of spew fillet surface area. Lower viscosity adhesive is able to produce fillet in different mass variants, whereas the application of small amount of high viscosity adhesive will not produce spew fillet.
2. The analysis of colour and consistence of adhesive layer following curing process provides initial quality control of adhesive joints.
3. Dimensional and shape accuracy depends on a number of factors, such as, the repeatability of joint forming conditions, the person producing the joint as well as technological factors.

Conducted tests reveal that it is both the type of adhesive and its amount which contribute to adhesive layer thickness. In the majority of instances the increase of adhesive composition mass was invariably accompanied by increased adhesive layer thickness; however, a correlation was evidenced concerning a certain amount of adhesive for which differences between adhesive layer thicknesses are diminishable. In addition, the viscosity of the adhesive was shown to influence the discrepancies in adhesive layer thickness in different adhesive composition mass variants. This factor was less noticeable in the case of highest viscosity adhesive. This may result from higher repeatability of measurement results following curing as compared to lower viscosity adhesive.

4. In the majority of analysed instances the increase of the mass of adhesive composition resulted in increased tensile-shear strength, moreover, the volume of increase is contingent on the type of adhesive.
5. A positive effect of spew fillet on adhesive joint strength was noted. The strength was, moreover, higher as the fillet surface area increased. It is, therefore, recommended that the fillet be included unless the technological considerations require its removal following cure.

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