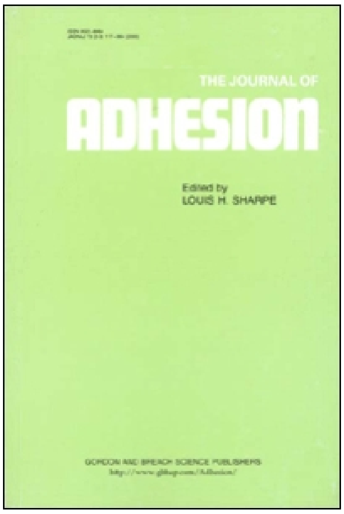

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A Study of Adhesive/Material/Surface Interactions for Galvanized Steel Sheet Assemblies

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The mechanical strength of bonded joints depends on a large number of factors, among which the adhesive-material-surface combination plays a dominant role. The analysis of the exact roles of these different factors during short destructive tests is delicate due to the complexity of the system. The use of a relatively simple experimental technique, the tensile test, has allowed us to perform a large number of tests and to apply a statistical analysis to the results. For galvanized steel sheets we have thus been able to specify the relative importance of each parameter in the bonding phenomenon and to indicate the interactions between these parameters, particularly for the material and surface condition pair.

KEY WORDS Adhesive joint, adhesive/material/surface interactions, bonded assembly, galvanized steel, statistical analysis, surface preparation.

I INTRODUCTION

The mechanical strength of bonded structures is usually evaluated by destructive tests which provide values of failure strength. There are a large number of test methods in use, indicating that no single test provides sufficient information to characterise a bonded joint completely. For example, one of the more popular tests, the shear test on a single lap joint specimen[1] yields an apparent average stress which does not relate to the actual stress distribution along the joint; in fact, several studies have shown that the stress reaches a maximum at the joint extremities.[2-10] The peel test[11] and the cleavage test[12] are characterised by the ratio of load at failure to the specimen width and thus do not take account of the area actually under stress during the test. The three point bending test[13] allows the determination of some interfacial characteristics but the testing conditions must be carefully selected.

In this study we have selected a tensile test based on the use of a small stud. In this type of test, as in many others, the measured stress is a mean stress because the stress state in the joint is certainly neither uniform nor uniaxial. In fact, the stress is maximum at the edges were the joint is simultaneously stressed in tension and flexure.[14] Nevertheless, this test is well reproducible and due to its simplicity, it allows a large number of tests to be performed. Thus, this approach has permitted a statistical analysis to be introduced in order to determine the relative importance of the parameters, adhesive-material-surface, on the bonding of sheets of galvanized steel as well as to detect the interactions which exist between these different parameters.

II MATERIALS AND EXPERIMENTAL TECHNIQUES

Three materials (substrates) have been studied: cold-rolled steel (B1), hot galvanized steel (B2) and annealed galvanized steel (B3); eight different material surface preparations were employed:

- degreasing by trichlorethane (C1): this was performed by rubbing the surface with a cotton swab, in order to remove greases, stains and foreign bodies from the surfaces. All materials were degreased in this way which therefore serve as both a reference and as a preparation for subsequent treatments
- treatment with sodium hydroxide: two treatments with NaOH were employed in order to remove the aluminium-rich surface

film on hot galvanized sheets:
1) NaOH, 0.5 M at 20°C for 5 minutes (C2)
2) NaOH, 2 M at 60°C for 10 seconds (C3)

- chemical etch: a sulphuric acid etch (H_2SO_4; 1 M) at 20°C for 13 minutes (C4) was used to clean the surface thoroughly
- treatment with parcodine: a treatment with a 20 vol.% solution in water of parcodine 120 (from Sté Continental Parker) was performed at 20°C for 13 minutes (C5)
- polishing: in order to examine the influence of the surface roughness, a polishing using silicon carbide abrasive papers of particle sizes 100 (140 μm) (C6), 400 (35 μm) (C7) and 1000 (18 μm) (C8) was carried out under water.

Three epoxy structural adhesives of different viscosities were studied:

Redux 609 (A1), AV118 (A2) and AV138 + HV998 (A3), all supplied by Ciba Geigy. The experimental method consisted of bonding aluminium alloy studs (Figure 1), of 6 mm^2 surface area, onto the substrates described previously (2 cm × 2 cm plates, 1.5 mm thick). Then, after the polymerization of the adhesive and the aging of the joint for 12 hours, tensile tests were carried out on a Sebastian 101 machine (load capacity 80 MPa), (Figure 2) at constant speed (1.2 mm/min). For each measurement eight tests were performed.

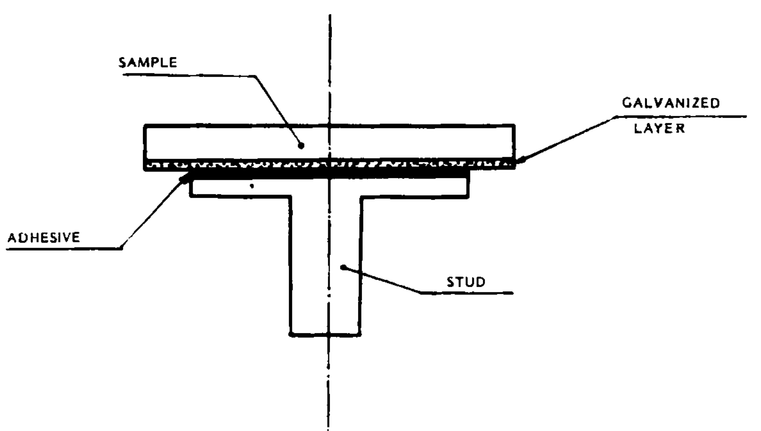

FIGURE 1 Diagram of the stud-sample assembly.

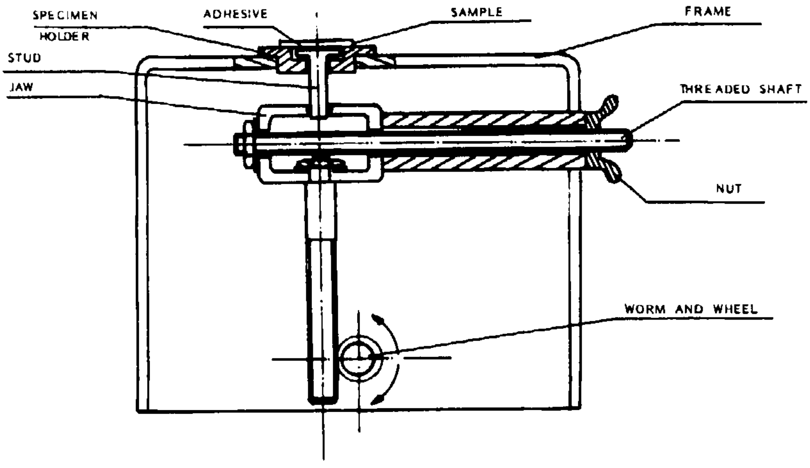

FIGURE 2 Schematic section through tensile testing machine.

III EXPERIMENTAL RESULTS

Results are presented as averages with standard deviation in Table I. Given the number of variables, the results in this form do not allow the influence of the different factors on the joint strength to emerge. We have therefore used an analysis of the variance[14] in an experimental plan with three controlled factors (A, B, C) with repetition. A is thus the adhesive factor, B the material factor and C the surface treatment factor (cf. Section II). Taking account of the description of the experimental study in the previous section, the number of modalities for the factor A is $r = 3$, for the factor B, $t = 3$ and for C, $u = 8$. The number of repetitions (number of tests for each measurement) is equal to 8.

The different terms in the analysis have been grouped in Table II, where, for each factor taken separately and for the various possible combinations, are presented:

- S.S. (square sum): the sum of squares for each factor
- $(n - 1)$: the number of degrees of freedom
- M.S. (mean square): the variance of the system M.S.$(A) =$ S.S.$(A)/n - 1$

TABLE I
Average value in MPa and standard deviation of the failure stress for all assemblies; the upper value represents the average and the lower value the standard deviation

A	*B*	*C* *C*1	*C*2	*C*3	*C*4	*C*5	*C*6	*C*7	*C*8	*Xi*...
	*B*1	25, 86 2, 20	20, 00 3, 09	17, 38 2, 60	25, 16 1, 34	18, 41 2, 10	25, 46 1, 20	24, 90 1, 75	26, 96 0, 84	
*A*1	*B*2	16, 52 2, 16	21, 60 2, 41	17, 27 1, 84	20, 20 1, 02	20, 35 2, 70	19, 86 1, 93	24, 18 1, 62	25, 13 2, 40	21, 42
	*B*3	22, 96 1, 77	21, 13 1, 46	20, 73 1, 80	21, 36 1, 22	16, 87 1, 95	22, 78 1, 85	18, 05 1, 87	20, 88 1, 35	
	*B*1	34, 10 1, 15	32, 43 1, 10	30, 98 2, 20	33, 02 1, 06	31, 88 1, 30	33, 87 1, 83	32, 91 1, 53	35, 50 1, 02	
*A*2	*B*2	28, 77 1, 58	33, 52 1, 34	29, 53 1, 57	31, 98 1, 85	32, 33 1, 04	29, 76 0, 96	33, 53 2, 27	33, 98 1, 66	30, 73
	*B*3	34, 77 2, 01	31, 30 1, 78	27, 60 1, 30	32, 33 1, 80	13, 66 1, 54	32, 87 0, 93	17, 06 0, 91	29, 83 1, 80	
	*B*1	18, 06 1, 23	16, 48 1, 15	14, 00 1, 00	17, 82 1, 41	14, 68 0, 58	17, 85 1, 10	17, 00 1, 70	20, 50 1, 10	
*A*3	*B*2	4, 70 0, 71	11, 91 1, 85	8, 23 0, 60	11, 91 1, 24	11, 83 0, 98	9, 50 0, 62	12, 97 9, 72	13, 02 1, 41	12, 22
	*B*3	12, 06 1, 03	10, 11 0, 78	7, 15 0, 87	10, 40 0, 73	6, 36 0, 51	11, 22 1, 10	6, 40 0, 50	8, 83 0, 40	
	X.k.	22, 00	22, 07	19, 20	22, 60	18, 49	22, 58	20, 78	23, 84	

TABLE II
Summary of the analysis (Fisher Test)[14]

Effect of	S.S.	$n-1$	M.S.	*Fc*	F_α $\alpha = 0, 01$
A	3289530	2	1644765	5966	4, 61
B	282037	2	141018	511	4, 61
C	168529	7	24075	87	2, 79
AB	83903	4	20975	76	3, 48
AC	53084	14	3791	13	2, 19
BC	299189	14	21370	77	2, 19
ABC	107319	28	3832	13	1, 86
E	138939	504	275		
Total	4422533	575			

- Fc: the calculated Fisher value $Fc(A) = \text{M.S.}(A)/\text{M.S.}(E)$ with M.S.(E) the residual variance
- F_α: the Fisher value extracted from standard tables (Fischer distribution) for a given confidence level $1 - \alpha$[15]

From the Fisher values, the significance of the influence of a factor on the phenomenon under study may be revealed. Tests of significance are carried out by making comparisons between values of Fc and F_α; the factor thus plays a significant role in the experimental results if Fc is greater than F_α (Table III).

The analysis thus indicates that all the factors A, B and C and their interactions are significant at the 1% level: this result is explained by the very low error value which seems to indicate that the methodology of the test is well controlled. In referring to the break-down of the sum of the squares it may be noted that in our experimental conditions, the part due:

- to factor A represents 74% of the total
- to factor B represents 7% of the total
- to factor C represents 4% of the total

The sum of the squares of the factors A, B, C and BC therefore represent about 92%, the error representing 4%; the 4% which remains represents the interactions AB, AC and ABC, indicating the minor importance of these three interactions. the factor A is therefore by far the most important followed by the factors B and C and the interaction BC. This analysis has thus revealed:

- that for the AV118 adhesive, failure loads are on average 30% higher than those for Redux 609, and 60% higher than those for

TABLE III
Tests of significance

	F_c		F_α
M.S.(A)/M.S.(E) =	5966	>	4,61
M.S.(B)/M.S.(E) =	511	>	4,61
M.S.(C)/M.S.(E) =	87	>	2,79
M.S.(AB)/M.S.(E) =	76	>	3,48
M.S.(AC)/M.S.(E) =	13	>	2,19
M.S.(BC)/M.S.(E) =	77	>	2,19
M.S.(ABC)/M.S.(E) =	13	>	1,86

AV138 + HV998 irrespective of the surface preparation for the three considered substrates

– that for the chosen adhesives and bonding conditions, cold-rolled steel failure loads are 14% higher than those for galvanized steel and 12% higher than those for galvannealed steel.

The role of the surface treatments may be emphasised by plotting graphically in a decreasing order the evolution, for a given substrate, of the failure stress *versus* the type of surface treatment for the different tested adhesives.

We thus observe (Figures 3, 4 and 5) that, for a given substrate, the relative effectiveness of the surface treatments is practically independent of the adhesive. On the other hand, the ranking of the effectiveness of the surface treatments varies with the different substrates thus confirming the existence of an interaction between the substrate and the surface treatment.

IV CONCLUSION

Measurements of bond strength from tensile tests with studs appeared to be well reproducible and therefore to be able to

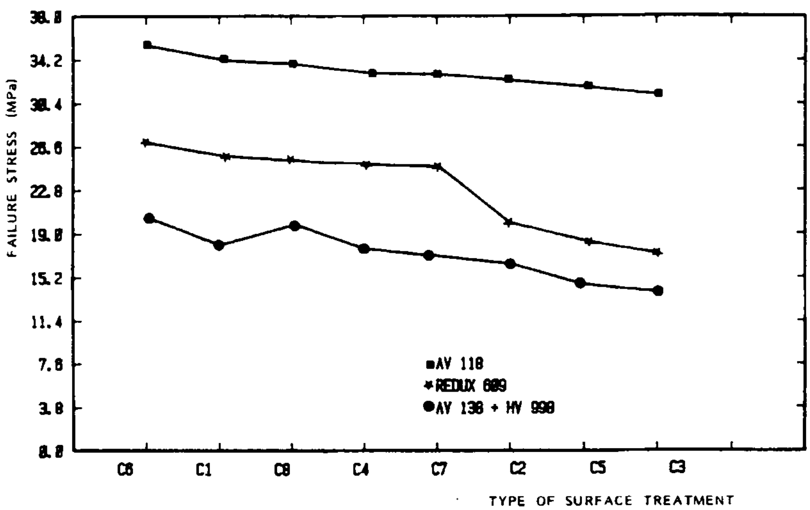

FIGURE 3 Variation of failure strength *versus* surface treatments for different adhesives in the case of cold rolled steel.

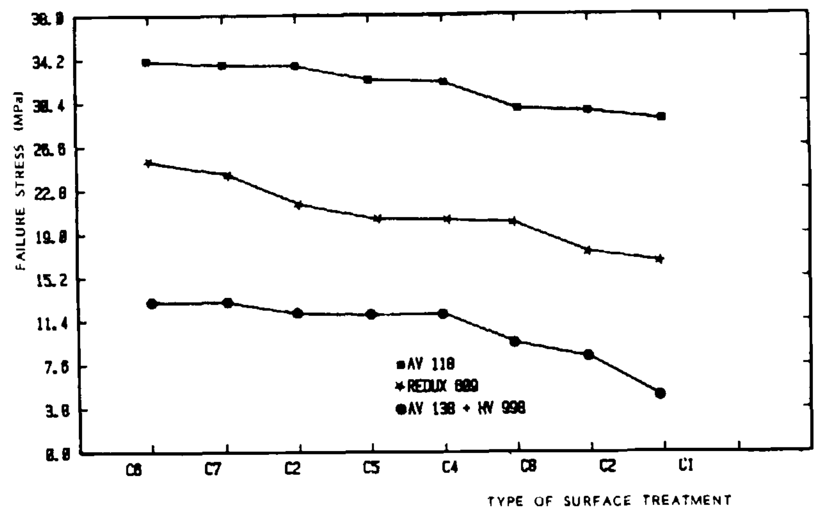

FIGURE 4 Variation of failure strength *versus* surface treatments for different adhesives in the case of galvanized steel.

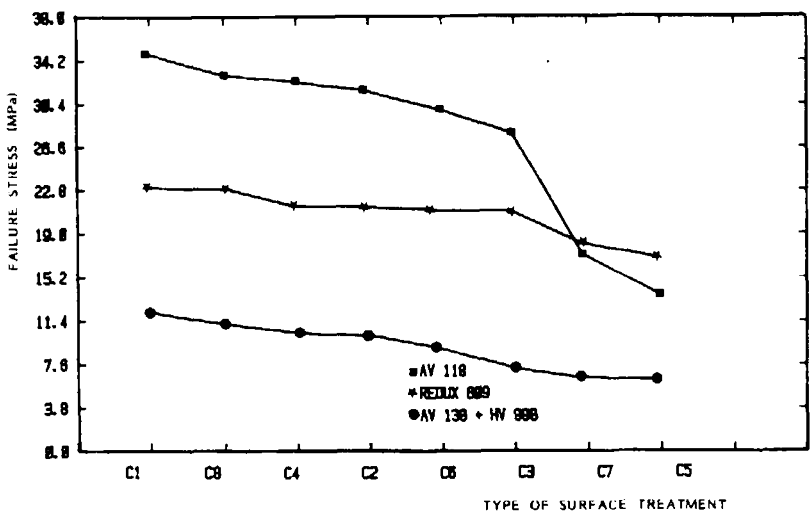

FIGURE 5 Variation of failure strength *versus* surface treatments for different adhesives in the case of galvannealed steel.

characterize the influence of factors such as the nature of substrate materials and adhesives or surface pretreatments on bond properties.

A large number of tests were performed, the results of which were treated by statistical analysis. This analysis showed that, despite the dominating influence of the adhesive on the joint behaviour, the role of the substrate material and that of the surface treatment could also be characterized. Moreover, a strong interaction between the nature of the substrate material and the type of surface treatment was also observed. Due to this interaction, synergistic effects can be obtained, resulting in the obtainment of bond strengths on galvanized steels similar to those obtained with cold rolled steels. The failure criterion based on this kind of tensile test, if not sufficiently rigorous to establish calculations for adhesively bonded structures, seems to be very useful in order to determine the influence of different parameters such as the surface preparation on the quality of the bond. The statistical approach proved to be very efficient in showing the role of the different parameters and their interactions and also the consistency of the methodology given the low value of the residual variance.

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