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**The Use of Statistical Methods for Evaluating
Durability of Adhesively Bonded Joints**

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ABSTRACT

This report presents a compilation of case studies that demonstrate the use of statistical methods in evaluating durability data generated in high humidity weathering conditions and accelerated laboratory tests. A number of case studies are based on data supplied by industrial organisations within the United Kingdom. The remaining case studies utilise data specifically generated within the experimental programme to evaluate key parameters (e.g. interaction between temperature and humidity) that influence long-term durability of adhesively bonded structures. Factors considered in the report include: (i) test method; (ii) surface treatment; (iii) adhesive type; (iv) processing variables (time, temperature and pressure); and (v) environmental conditions.

Statistical analysis has been used to determine the effect of high humidity weathering conditions (i.e. 95% humidity with the temperature cycled between 42 °C and 48 °C) on the performance of zinc and organic coated steel joints bonded with a range of adhesives (polyurethane, acrylic and epoxy). The data, supplied by British Steel Plc, was for a 12 month exposure period. Data, supplied by British Aerospace (Sowerby), relating to the lap shear and thick adherend shear test (TAST) methods conducted on two adhesives over the temperature range of -67 °F (-55 °C) to 350 °F (177 °C) have been compared in order to determine relative sensitivity and data variability for the two methods. A third case study examines data supplied to the programme by Alcan International Limited, in which a full factorial analysis is used to determine the optimum combination of processing variables for long-term durability performance.

The report concludes with two case studies based on durability data generated within the programme. The first demonstrates the use of a "Design of Experiment" approach to maximise information from an experiment, and simultaneously minimise the impact of spurious non-experimental, or "noise" factors, during the execution of the experiment. The case study concludes with an analysis of the results of the controlled experiment. The final case study considered in this report examines the interaction of temperature and humidity on residual strength of titanium alloy joints bonded with an epoxy structural adhesive. Single-lap joints were exposed to nine combinations of temperature (3 levels) and humidity (3 levels). A full factorial analysis has been carried out on the data to determine empirical relationships between the various factors of time, temperature and humidity.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
Head of Centre for Materials Measurement and Technology.

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FOREWORD

In a previous report (“Statistical Analysis of Durability Data” NPL Report CMMT(A) 202), a number of statistical analysis tools were used to identify and quantify the effect that critical factors and their interactions have on environmental resistance of adhesively bonded joints. These techniques enabled the derivation of empirical relationships to be determined for both a full and partial sets of data and provided a measure of “goodness” of fit between predictive models and durability data. This report extends this work through a series of case studies that demonstrate the use of statistical methods in evaluating durability data generated under high humidity weathering conditions and supplied by members of the industrial advisory group (IAG) and accelerated laboratory tests. The remaining case studies utilise data specifically generated within the experimental programme to evaluate key parameters (e.g. interaction between temperature and humidity) that influence long-term durability of adhesively bonded structures. Factors considered in the report include: (i) test method; (ii) surface treatment; (iii) adhesive type; (iv) processing variables (time, temperature and pressure); and (v) environmental conditions.

Statistical analysis has been used to determine the effect of high humidity weathering conditions (i.e. 95% humidity with the temperature cycled between 42 °C and 48 °C) on the performance of zinc and organic coated steel joints bonded with a range of adhesives (polyurethane, acrylic and epoxy). The data, supplied by British Steel Plc, was for a 12 month exposure period. Data, supplied by British Aerospace (Sowerby), relating to the performance lap shear and thick adherend shear test (TAST) methods conducted on two adhesives over the temperature range of -67 °F to 350 °F have been compared in order to determine relative sensitivity and data variability for the two methods. A third case study examines industrial data supplied to the programme by Alcan International Limited, in which a full factorial analysis is used to determine the optimum combination of processing variables for long-term durability performance.

The report includes two case studies based on durability data generated within the programme. The first demonstrates the use of a “Design of Experiment” approach to maximise information from an experiment, and simultaneously minimise the impact of spurious non-experimental, or “noise” factors, during the execution of the experiment. The case study concludes with an analysis of the results of the controlled experiment. The final case study considered in this report examines the interaction of temperature and humidity on residual strength of titanium alloy joints bonded with an epoxy structural adhesive. Single-lap joints were exposed to nine combinations of temperature (3 levels) and humidity (3 levels). A full factorial analysis has been carried out on the data to determine empirical relationships between the various factors of time, temperature and humidity. The order in which the case studies are discussed is presented below:

Case Study 1: Statistical Analysis of the Long-Term Durability of Adhesively Bonded Joints with Zinc and Organic Coated Steels and Various Surface Treatments.

Case Study 2: Statistical Comparisons of Lap-Shear and Thick-Adherend Shear Test Data.

Case Study 3: Statistical Analysis of Thick Adherend Results.

Case Study 4: Aluminium Vehicle Treatment Results.

Case Study 5: Temperature-Humidity Interaction.

The research discussed in this report forms part of the DTI funded project on “Performance of Adhesive Joints - Combined Cyclic Loading and Hostile Environments”, which aims to develop and validate test methods and environmental conditioning procedures that can be used to measure parameters required for long-term performance predictions. This project is one of three technical projects forming the programme on “Performance of Adhesive Joints - A Programme in Support of Test Methods”.

Case Study 1

**Statistical Analysis of the Long-Term Durability of
Adhesively Bonded Joints with Zinc and Organic Coated
Steels and Various Surface Treatments**

1. INTRODUCTION

The first case study relates to work carried out by British Steel Plc on “The Effect of Environment and Temperature on the Long-Term Durability of Adhesively Bonded Joints in Zinc and Organic Coated Steels” (Report No. EUR 17998 EN) [1]. From this comprehensive research programme, two particular experiments were selected for further analysis:

Experiment 1 centred on the effects of **various adhesives** on lap shear strength with exposure time for both uncoated and coated mild steel joints.

Experiment 2 centred on the effects of **various surface treatments** on lap shear strength with exposure time for both uncoated and coated mild steel joints.

Single-lap joint specimens, unstressed, were subjected to high humidity weathering conditions (i.e. 95% humidity with the temperature cycled between 42 °C and 48 °C) for periods up to 12 months. “The environment is classed as a severe weathering cycle and is claimed that the results obtained can give an indication of the likely service performance of bonded joints for periods of up to 10 years. It has been successfully used to give an indication of the influence of substrates, surface treatments and adhesives on durability, where systems of inherently poor durability are frequently highlighted in the first two months of weathering [1]”. Batches of specimens were removed on a monthly basis and tested to failure under standard laboratory conditions.

This case study utilises statistical procedures to examine the significance of the various factors involved in the two experiments.

2. EXPERIMENT 1 (ADHESIVES/COATING/TIME)

The experiment investigated the influence of exposure time on single-lap joints constructed from uncoated and metallic (iron zinc alloy) coated mild steel with a range of different adhesives. The response measure used was the lap shear strength (N/mm²). The experimental factors and levels were as follows:

- A.** Adhesives - Polyurethane, Acrylic, Epoxy A, Epoxy B, Epoxy C and Epoxy D.
- C.** Coating - Uncoated, Iron Zinc Alloy.
- T.** Time - 0 to 12 months.

Three specimens were evaluated per test condition. The data is shown in Appendix 1.1.

2.1 ANALYSIS OF VARIANCE (ANOVA)

Details of the analysis for all the data is shown in the Analysis of Variance (ANOVA) table in Appendix 1.2. This table shows the contribution to the variation in the experimental results due to the different factors and their interactions. It can be seen from the SIG LEVEL column, that Time (**T**), Adhesives (**A**) and Coating (**C**) all make a significant contribution to the variation (NB. The smaller the SIG LEVEL the more significant the

effect). Hence, the factors themselves are significant. In addition, the table shows that the interaction between the factors is also significant and hence the interaction of Time and Adhesives (**TA**), Time and Coating (**TC**) and Adhesive and Coating (**AC**) are all significant effects.

2.2 ANALYSIS OF MEANS (ANOM)

Analysis of Means charts (Appendix 1.2 and 1.3) provide a graphical analysis of the main effects and interaction effects, thus enabling a greater insight than can be seen in the ANOVA table. The chart in Appendix 1.2 shows that the average effect of each of the three main factors **T**, **A** and **C** over their respective levels. The overall impact of Time is clearly demonstrated over the full range from 0 to 12 months with a significant strength loss over the first two months, followed by a levelling off, but still downward effect over the period 3 to 12 months. The Adhesive chart shows that epoxy C adhesive has by far the highest strength overall and the Polyurethane and Acrylic adhesives the lowest overall strengths. The Coating chart shows that the specimens with a metallic (i.e. Iron Zinc Alloy) coating have a significantly higher strength compared with the uncoated specimens.

The chart in Appendix 1.3 **shows an average effect for each of the interactions TA, TC and AC**. It can be seen from the **TA** chart that the effect of adhesives is not the same at each of the time intervals; noticeably 0 to 1 month. The **TC** chart shows a similar effect. The change from uncoated to the metallic coating is markedly different over the same period compared with the following months. The **AC** chart shows that the change in surface treatment from uncoated to metallic coated does not have the same effect for each of the adhesives. This is most marked for adhesive B, which shows a large effect, and adhesive D where there is no significant effect.

2.3 UNCOATED VERSUS METALLIC COATED ANALYSIS

The previous analyses have shown that there is a significant coating effect with the metallic coating generally giving higher lap shear strength results. In addition, there is a significant interaction between Coating and Time, and Coating and Adhesives. These more subtle effects are investigated through the aid of the charts in Appendix 1.4. In each of the three charts (with Time at 0 months, 3 months and 12 months), the box plots show a visual comparison of the results for uncoated and metallic coated joints for each adhesive. The more formal test of significance is included in the table, which includes the Bartlett test of variation and the ANOVA test of averages.

At 0 months, only adhesive A shows any significant difference in average strength for uncoated and metallic coated specimens with the former being higher. In addition, all the standard deviations are all consistent with each other.

At 3 months, all the adhesives with the exception of adhesive D show a significant difference in average strength for the two types of coating with the metallic coating resulting in higher average strengths. Again, all the standard deviations are all consistent with each other.

At 12 months, epoxy adhesives B and C, and Polyurethane and Acrylic adhesives show a significant difference in average strength for the two types of coating. Again, the metallic coating gives higher average strengths. The standard deviations associated with the epoxy adhesives, A and B, and Polyurethane and Acrylic differ for uncoated and coated specimens.

2.4 ADHESIVE ANALYSIS

The previous analyses have shown that there is a significant adhesive effect. The charts in Appendix 1.5 seek to answer the questions “Which adhesives are different from the others?” and “Are differences maintained across coatings and for different time periods?”.

Three graphical representations of the data for 0, 3 and 12 months are presented in Appendix 1.5:

- Top:** Box plots comparing the average joint strengths for each adhesive system and coating.
- Middle:** A table showing the significance of the effects via the Bartlett and ANOVA tests.
- Bottom:** A table assessing the significance of each adhesive compared with each other.

The formal test of significance using Duncan’s Multiple Range procedure, produces a ranking of the adhesives, together with statements about which adhesives are the same, and which are different.

At 0 months: Both uncoated and metallic coated joints show a significant difference in average strength across the adhesives. The standard deviation is different for uncoated specimens (middle table in Appendix 1.5). The “Significance of Adhesives” table shows that for uncoated joints, adhesives A, B and D are indistinguishable from each other. All other comparisons between adhesives are significant with Polyurethane being the lowest and adhesive C the highest. For metallic coated joints, Polyurethane and Acrylic bonded systems behave the same, as do joints bonded with either A, B or D epoxy adhesives. All other comparisons are significantly different, with Polyurethane / Acrylic being the lowest and adhesive C the highest.

At 3 months: Both uncoated and metallic coated joints show a significant difference in average strength for the different adhesives. The standard deviation is different for uncoated specimens. The “Significance of Adhesives” table shows that for an uncoated substrate, the following adhesive systems behave the same:

- Epoxy B, Polyurethane and Acrylic
- Polyurethane, Acrylic and EpoxyA
- Epoxies A and D
- Epoxies D and C

All other comparisons are significantly different with adhesives B/Polyurethane/Acrylic being the lowest and adhesives D/C the highest. For metallic coated adherends, Epoxy D, Polyurethane and Acrylic adhesives behave the same, as do adhesives B and C. All other comparisons being different (**Lowest** - D/Polyurethane/Acrylic; **Highest** - B/C).

At 12 months: Both uncoated and metallic coated joints continue to show a significant difference in average strength across the adhesives. The standard deviation is different for uncoated specimens. It can be seen in the “Significance of Adhesives” table that for uncoated joints, Epoxy B, Polyurethane, Acrylic and Epoxy A adhesives behave the same, as do adhesives A and D, and adhesives D and C. All other comparisons are significantly different (**Lowest** - B/Polyurethane/Acrylic/A; **Highest** - D/C). For metallic coated joints, Polyurethane, Epoxy D, Epoxy A and Acrylic adhesives behave the same, as do adhesives C and B. All other combinations are significantly different (**Lowest** - Polyurethane/D/A/Acrylic; **Highest** - C/B).

2.5 WEATHERING TIME ANALYSIS

It has been previously shown that there is a significant weathering time effect. This is investigated further in Appendix 1.6. The two charts in Appendix 1.6 show the change in joint strength for both uncoated and coated specimens for all six adhesive systems. Unconditioned (i.e. 0 months) and 12 month conditioned data are compared.

For uncoated joints, all adhesives showed significant differences in joint strength between the unconditioned (Time 0 months) and conditioned (Time 12 months) states. Adhesive B suffered the greatest average loss at 100% with adhesive C suffering the smallest loss at 70%.

For coated joints, adhesives A, C and D showed significant differences between Time 0 and Time 12 months with adhesive D suffering a 54% loss in strength. Epoxy B, Polyurethane and Acrylic adhesives showed no significant reduction in strength. The lowest measured reduction was for adhesive B with an average loss of 12%.

An interesting observation from the analyses is that adhesive B, which was the worst performing adhesive for uncoated joints, was the best performing adhesive with the metallic coating. This shows a feature of the interaction between factors highlighted earlier.

2.6 CONCLUSIONS FROM EXPERIMENT 1

- The analysis approach of ANOVA, ANOM, Box Plots, Bartlett tests and Duncan’s Multiple Range test has provided a comprehensive methodology for assessing the statistical significance of factors included in the experiment.
- The analysis has shown that the factors of Time, Coating and Adhesive and their interactions were all significant.

- General statements of significance:
 - a) Coating the mild steel adherends with iron zinc alloy generally improved environmental resistance. Although the shear lap strength for both uncoated and coated joints decreased with exposure time, the loss of strength over 12 months was more marked for the uncoated specimens (86% vs 32% strength loss). The shear lap strength was initially higher (i.e. unconditioned state) for the uncoated specimens, but with increasing exposure time the shear lap strength of the coated specimens exceeded the uncoated specimen strength.
 - b) There was a significant exposure time effect with most reduction in strength occurring in the first 2 months of exposure.
 - c) Epoxy C adhesive gave the highest results overall. This overall performance held good for both uncoated and coated specimens over each time interval, thus proving the adhesive to be very robust.
 - d) Adhesive Epoxy B showed a very significant improvement in environmental performance when the adherends were coated in the iron zinc. The metallic coated joints suffered a 12% loss in strength over 12 months exposure compared with almost a 100% loss in strength experienced by uncoated joints over the same period. Environmental performance of Epoxy B/ metallic coated adherends matched that of adhesive C.
- Statements of significance are based on the experimental data. When a comparison is deemed **not** significant, it means that any real difference, if it exists, is unable to be determined. The discriminating ability is generally not a problem in determining important main effects, but it could be a problem in untangling interaction effects. This may be because the sample size, or replications, used in the experiment is too small, and/or the experimental error is too large. Discrimination between significant effects and non-significant effects can only be improved by either increasing sample size or reducing experimental error.

3. EXPERIMENT 2 (SURFACE TREATMENT/COATING/TIME)

Experiment 2 investigated the influence of exposure time of uncoated and iron zinc alloy (metallic) coated mild steel bonded with Epoxy A (same adhesive used in Experiment 1) for a range of surface treatments. The experimental factors and levels were as follows:

- S.** Surface Treatment - Degreased, Phosphated, Chromated.
- C.** Coating - Uncoated, Iron Zinc Alloy.
- T.** Time - 0 to 12 months.

Three specimens were evaluated at each of the test conditions. The data is shown in Appendix 2.1.

3.1 ANALYSIS OF VARIANCE (ANOVA)

Details of the analysis for all the data is shown in the Analysis of Variance (ANOVA) table in Appendix 2.2. This table shows the contribution to the variation in the experimental results due to the different factors and their interactions. It can be seen from the SIG LEVEL column, that Time (**T**), Surface (**S**) and Coating (**C**) all make a significant contribution to the variation. Hence, the factors themselves are significant. In addition, the table shows that the interaction between the factors is also significant and hence the interaction of Time and Surface (**TS**), Time and Coating (**TC**) and Surface and Coating (**SC**) are all significant effects.

3.2 ANALYSIS OF MEANS (ANOM)

Analysis of Means charts (Appendix 2.2 and 2.3) provide a graphical analysis of the main effects and interaction. The chart in Appendix 2.2 shows that the average effect of each of the three main factors **T**, **S** and **C** over their respective levels. The overall impact of Time is clearly demonstrated over the full range from 0 to 12 months with a significant strength loss over the first two months, followed by a levelling off, but still downward effect over the period 3 to 12 months. The Surface Treatment chart shows that the Chromated surface gives by far the highest strength overall and that Degreased and Phosphated surface treatments produce similar results. The Coating chart shows that the specimens with a metallic (i.e. Iron Zinc Alloy) coating have a significantly higher strength compared with the uncoated specimens.

The chart in Appendix 2.3 **shows an average effect for each of the interactions TS, TC and SC**. It can be seen from the **TS** chart that the effects of surface treatments is not the same at each of the time intervals; notably 0 to 1 month. The **TC** chart shows a similar effect. The change from uncoated to the metallic coating is markedly different over the same period compared with the following months. The **SC** chart shows that the change in surface treatment from uncoated to metallic coated does not have the same effect for each of the surface treatments. This is most marked when comparing the results of surfaces that have been chromated, where there is no difference between coating conditions, with those that have either been degreased or phosphate treated, where the differences are large and similar in magnitude between the two coating conditions.

3.3 UNCOATED VERSUS METALLIC COATED ANALYSIS

The previous analyses have shown that there is a significant coating effect with the metallic coating generally giving higher lap shear strength results. In addition, there is a significant interaction between Coating and Time, and Coating and Surface Treatment. These more subtle effects are investigated through the aid of the charts in Appendix 2.4. In each of the three charts (with Time at 0 months, 3 months and 12 months), the box plots show a visual comparison of the results for uncoated versus metallic coated joints for each surface treatment. The more formal test of significance is included in the table, which includes the Bartlett test of variation and the ANOVA test of averages.

At 0 months, only those specimens that have been degreased show any significance difference in average strength between the two coating conditions with the strength being

higher for the uncoated specimens. All the standard deviations are all consistent with each other.

At 3 months, only those specimens that have been phosphate treated show any significance difference in average strength between the two coating conditions. Again, all the standard deviations are all consistent with each other.

At 12 months, only those specimens that have been phosphate treated show any significance difference in average strength between the two coating conditions. The difference between the standard deviation for the two coating conditions is significant for those specimens that have been degreased.

3.4 SURFACE TREATMENT ANALYSIS

The previous analyses have shown that there is a significant surface treatment effect. The charts in Appendix 2.5 seek to answer the questions “Which surface treatments are different from the others?” and “Are differences maintained across coatings and for different time periods?”.

Three graphical representations of the data for 0, 3 and 12 months are presented in Appendix 2.5 (for further details see Section 1.5). The formal test of significance using Duncan’s Multiple Range procedure, produces a ranking of the surface treatments, together with statements about which adhesives are the same, and which are different.

At 0 months: Uncoated joints show a significant difference in average strength across the surface treatments. The standard deviation is different for uncoated specimens (middle table in Appendix 2.5). The “Significance of Surface Treatments” table shows that for uncoated joints, Degreased and Chromated results are indistinguishable from each other. All other comparisons between surface treatments are significant with the Phosphated results being the lowest and Degreased / Chromated the highest. For the coated specimens, there is no significant difference between the surface treatments.

At 3 months: Both uncoated and metallic coated joints show a significant difference in average strength for the different surface treatments. The standard deviation is different for uncoated specimens. The “Significance of Adhesives” table shows that for an uncoated substrate, the following surface treatments behave the same:

- Phosphated and Chromated
- Chromated and Degreased

All other comparisons between surface treatments are significantly different with Phosphated / Chromated being the lowest and Chromated / Degreased the highest.

For coated specimens, the following surface treatments behave the same:

- Degreased and Phosphated
- Phosphated and Chromated

All other comparisons between surface treatments are significantly different with Degreased / Phosphated being the lowest and Phosphated / Chromated the highest.

At 12 months: Both uncoated and metallic coated joints continue to show a significant difference in average strength across the adhesives. The standard deviations are all consistent with each other. It can be seen in the “Significance of Adhesives” table that for uncoated and coated joints, Degreased and Phosphated surface treatments behave the same. All other comparisons between surface treatments are significantly different with Degreased / Phosphated being the lowest and Chromated the highest.

3.5 WEATHERING TIME ANALYSIS

As previously shown, there is a significant weathering time effect. This is investigated further in Appendix 2.6. The two charts in Appendix 2.6 show the change in joint strength for both uncoated and coated specimens for all three surface treatments. Unconditioned (i.e. 0 months) and 12 month conditioned data are compared.

For uncoated joints, all surface treatments showed significant differences in joint strength between the unconditioned (Time 0 months) and conditioned (Time 12 months) states. Degreased surface treated specimens suffered the greatest average loss of strength (83%) with Chromated surface treated specimens suffering the smallest strength loss (41%).

For coated joints, both Degreased and Phosphated surface treatments showed significant differences between the unconditioned (0 months) and 12 months conditioning with the Phosphated treated specimens suffering a 45% reduction in strength. The Chromated surface showed no significant reduction in strength, although the measured reduction was 30%.

3.6 CONCLUSIONS FROM EXPERIMENT 2

- As with Experiment 1, the analysis approach of ANOVA, ANOM, Box Plots, Bartlett tests and Duncan’s Multiple Range test has provided a comprehensive methodology for assessing the statistical significance of factors included in the experiment.
- The analysis has shown that the factors of Time, Coating and Surface Treatment and their interactions were all significant.
- General statements of significance:
 - a) Coating the mild steel adherends with iron zinc alloy generally improved environmental resistance. Although the shear lap strength for both uncoated and coated joints decreased with exposure time, the loss of strength over 12 months was more marked for the uncoated specimens (60% vs 39% strength loss). The shear lap strength was initially higher (i.e. unconditioned state) for the uncoated specimens, but with increasing exposure time the shear lap strength of the coated specimens exceeded the uncoated specimen strength.

- b) There was a significant exposure time effect with most reduction in strength occurring in the first 2 months of exposure.
 - c) The Chromated surface treatment gave the highest results overall. This overall performance held good for both uncoated and coated specimens over each time interval. This proved to be a very robust surface treatment.
-
- Statements of significance are based on the experimental data. When a comparison is deemed **not** significant, it means that any real difference, if it exists, is unable to be determined. The discriminating ability is generally not a problem in determining important main effects, but it could be a problem in untangling interaction effects. This may be because the sample size, or replications, used in the experiment is too small, and/or the experimental error is too large. Discrimination between significant effects and non-significant effects can only be improved by either increasing sample size or reducing experimental error.

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ADDITIONAL READING

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APPENDIX 1.1

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APPENDIX 1.1

ORIGINAL DATA FROM BRITISH STEEL (REF. REPORT NO. EUR 17998 EN)

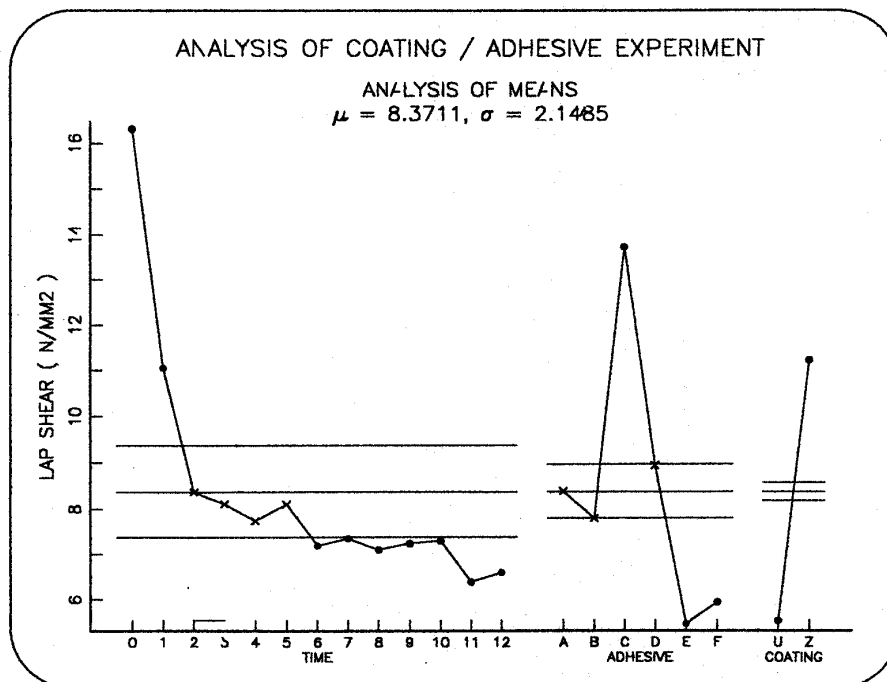
Adhesive	1.22mm uncoated MILD STEEL- (Degreased with Inhibisol)												
	Exposure time (months)												
	0	1	2	3	4	5	6	7	8	9	10	11	12
A	19.6	22.8	7.35	4.35	5.3	3.25	2.95	5.35	3.1	3.75	0.1	2.25	8.4
	20	22.8	10.4	5.7	4.1	6.7	0.1	4.35	7.9	1.1	0.2	3.05	1.2
B	19.9	11.3	7.35	6.4	3.4	0.85	3	4.65	2.9	4.2	0.1	2.5	0.5
	19.3	6.3	0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C	24.3	6.6	0.2	2	0.2	2.05	1.35	1.6	0.2	0.2	0.2	0.2	0.2
	16.5	8.9	0.1	0.1	0.1	0.45	0.1	0.1	0.1	0.1	0.1	0.1	0.1
D	25.5	16.6	12.7	10.8	9.6	11.3	1.2	6.95	7.1	8.9	0.1	8.5	6.95
	27.3	17.2	14.3	12.2	11.1	9.9	12.6	9.5	8.45	9.05	9.6	8.85	8.6
POLY	24.5	17.9	13	10.8	10.3	10.4	11.8	9	9.15	9.6	8.6	7.7	8
	17.6	10.9	7.9	11.6	11.6	10.7	12	9.55	4.5	9	9.35	6.35	4.25
ACRYLIC	17.2	8.65	11.3	11	3.65	9.15	8	10.3	7.3	7.15	8.5	9.3	5.35
	16	12.9	8.65	2.5	10.9	10.5	10.6	9.2	9.15	8.05	5.6	4.1	6.8
POLY	7.7	0.3	0.12	0.9	1.45	0.4	0.1	0.5	0.6	0.05	0.1	0.1	0.1
	8.25	0.35	0.3	2.65	2.1	0.8	0.2	0.65	0.4	1	0.2	0.2	0.2
ACRYLIC	8.45	0.1	1.8	0.65	1.45	0.25	0.15	0.1	2.25	0.3	0.1	0.1	0.1
	11	4.1	3.85	2.05	3.15	2.45	1.4	2.2	1.75	1	2.4	3.35	0.1
ACRYLIC	10.7	8.9	2.35	2.85	1.15	3.45	1.8	2	2.5	1.55	1.55	2.94	0.2
	12.8	2.5	3.05	2.65	3.6	2.55	1.95	1.8	2.25	0.7	1.65	2.45	0.1

Adhesive	1.1mm IRON -ZINC ALLOY coated steel (Degreased with Inhibisol)												
	Exposure time (months)												
	0	1	2	3	4	5	6	7	8	9	10	11	12
A	15.8	11.4	10.3	11.5	9.25	9.65	8.4	10.2	9.15	8.95	14.8	10.1	8.5
	15.9	12.1	9.35	9.6	9.7	9.1	11.2	10.4	8.35	9.65	11.5	9.5	9.5
B	15.1	8.7	10.7	11	9.9	11.2	9.85	9.4	10.8	9.7	10.5	10.3	9.5
	18.8	8.7	7.7	15.3	10.5	11.4	12.1	12.4	14.7	11.7	15.5	8.35	13.4
C	11.9	10.2	7.25	13.5	16.3	13.9	9.9	14.3	9.9	13.4	20	11.8	15.5
	18.6	15.9	14.8	16.3	16.2	16.4	9.5	10.3	14.7	14.6	14.4	10	14.6
D	21.6	22.8	16.7	15.8	16.8	15.5	14.8	14.8	14.8	14.1	14.7	15.1	14.3
	24.3	19.5	18.3	18.1	15.9	18.2	15.9	15.9	15.9	13.6	11.2	12.5	15.2
POLY	24.1	16.8	17.8	16.7	16.4	16.4	15.2	15.2	15.2	13.9	14.4	15	12.2
	15.9	12.1	10.8	8.15	12.3	8.7	8.35	3.25	9.9	11.2	3.6	3.7	10.3
ACRYLIC	17.8	18.6	5	3.7	9.25	12.1	11.3	13.2	10.9	3.1	10.6	7.2	3.2
	16.1	4.6	10.9	10.4	4.4	2.85	3.6	3.9	4.45	10.9	9.3	4.5	9.5
POLY	12	11.6	7.5	9.25	10.3	11.9	10.6	8.1	5.9	11.7	12	8.7	6.75
	8	9.35	8.1	10.2	10.3	11.4	9.3	9.15	9	11	12.1	7.2	6
ACRYLIC	11.8	8.75	10.7	9.7	11.1	12.9	10.8	10.7	8.75	9.55	6.75	11.1	10
	11.6	10.3	9.2	7.2	5.5	8.35	11.8	8	7.95	9.25	11.2	10.4	7.25
ACRYLIC	11.3	8.35	9.55	8.35	7.75	7	9.1	5.15	7.25	9.1	11.3	4.65	9.7
	10	9.25	11.8	7.8	3.4	9.15	7.6	12.2	8.3	9.3	10	7.75	11

APPENDIX 1.2

ADHESIVE / COATING / TIME EXPERIMENT**ANALYSIS OF VARIANCE**

SOURCE	DF	SS	MS	F RATIO	SIG. LEVEL
T TIME	12	3038.6	253.22	54.86	2.2E-16
A ADHESIVE	5	3419.8	683.97	148.17	0
C COATING	1	3787.3	3787.3	820.45	0
TA	60	767.23	12.79	2.77	5.4E-9
TC	12	759.26	63.27	13.71	5.6E-16
AC	5	1453.8	290.77	62.99	0
TAC	60	631.14	10.52	2.28	2.7E-6
ERROR (PURE)	312	1440.2	4.62		
TOTAL	467	15298	32.76		

ANALYSIS OF MEANS - MAIN EFFECTS

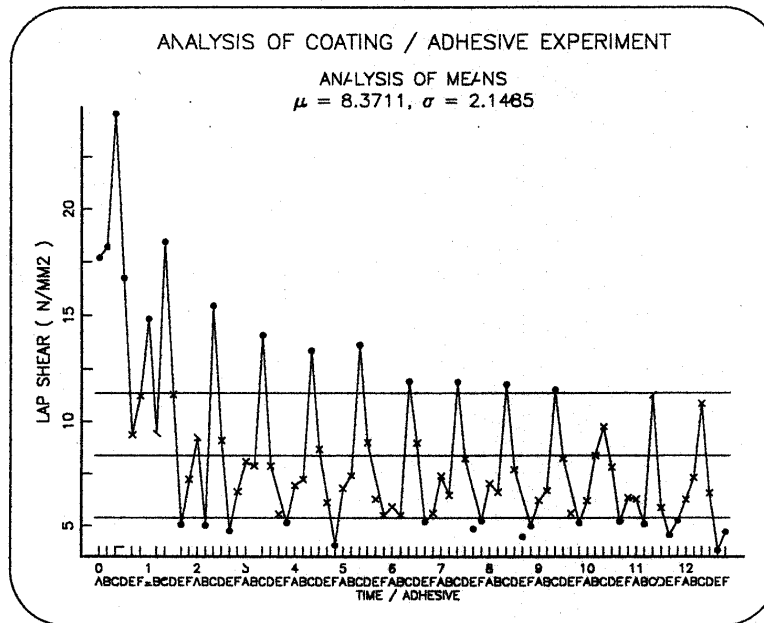
NOTES.

Adhesive E = Polythene ; Adhesive F = Acrylic; Others as per letter.
 Coating U = Uncoated ; Coating Z = Iron Zinc Alloy

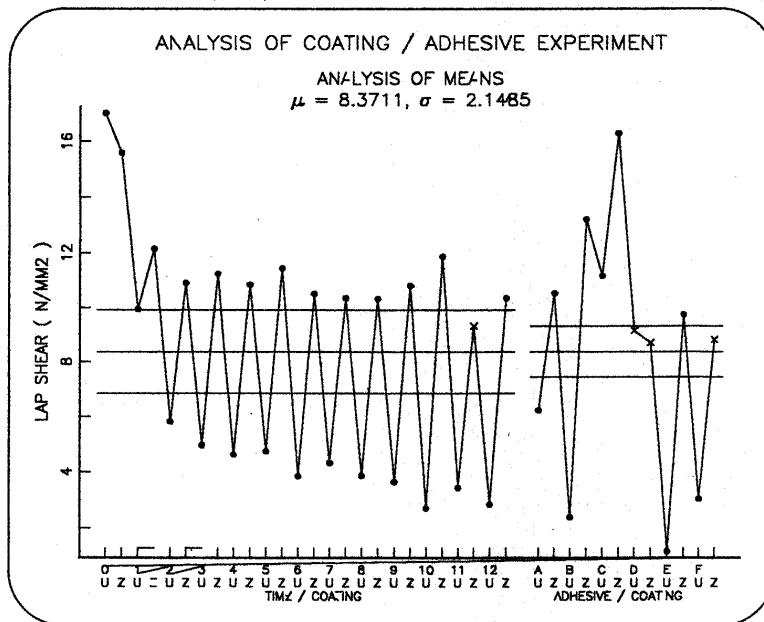
APPENDIX 1.3

ANALYSIS OF MEANS - INTERACTION EFFECTS

TIME / ADHESIVE (TA)



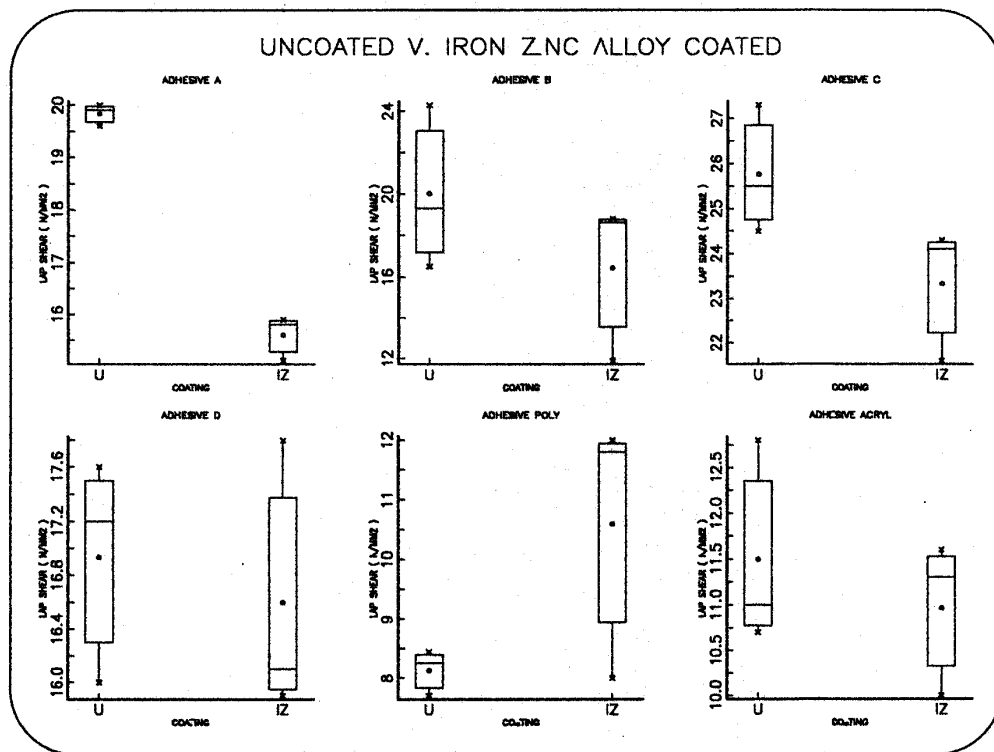
TIME / COATING (TC) ----- ADHESIVE / COATING (AC)



NOTES.

Adhesive E = Polythene ; Adhesive F = Acrylic; Others as per letter.
 Coating U = Uncoated ; Coating Z = Iron Zinc Alloy

APPENDIX 1.4

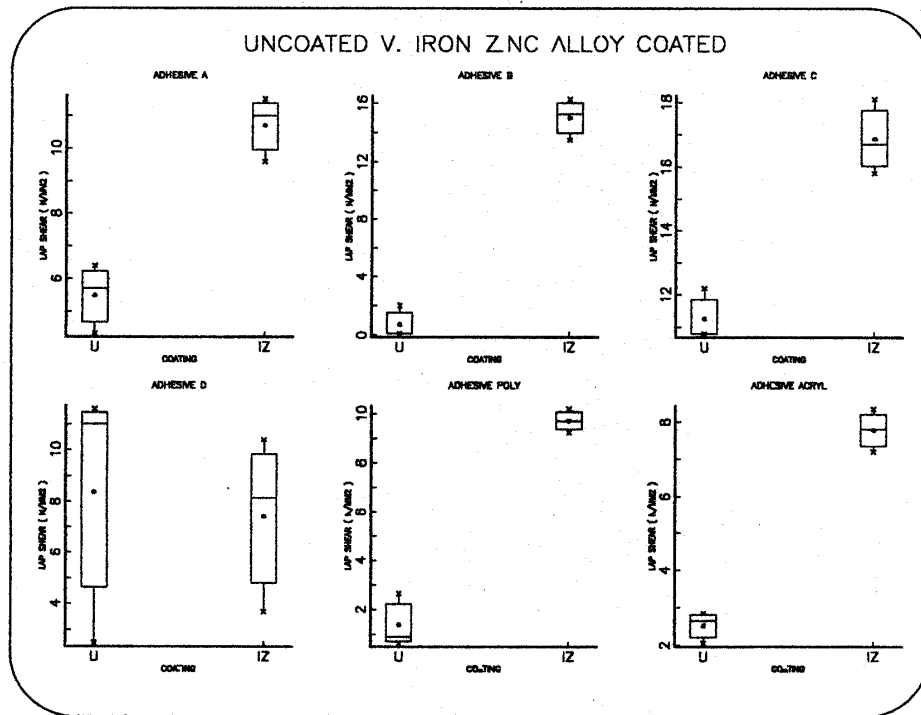
UNCOATED V. IRON ZINC ALLOY - (0 MONTHS)

ADHESIVE	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
A	19.83	0.21	15.6	0.44	SAME	DIFF
B	20.03	3.95	16.43	3.92	SAME	SAME
C	25.77	1.42	23.33	1.5	SAME	SAME
D	16.93	0.83	16.6	1.04	SAME	SAME
POLYTHENE	8.13	0.39	10.6	2.25	SAME	SAME
ACRYLIC	11.5	1.14	10.97	0.85	SAME	SAME

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 1.4 (CONT.)

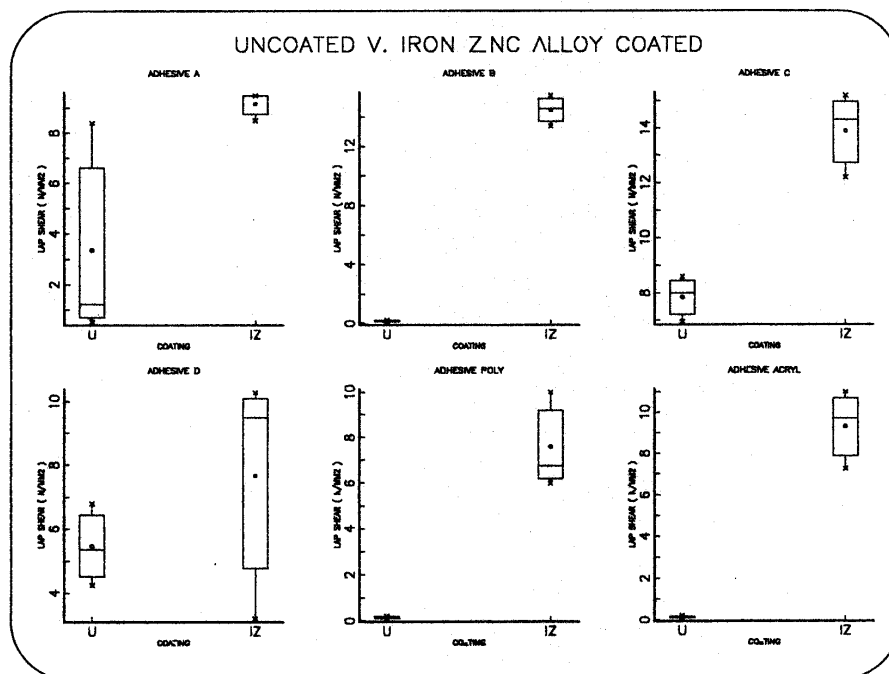
UNCOATED V. IRON ZINC ALLOY - (3 MONTHS)

ADHESIVE	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
A	5.48	1.04	10.7	0.98	SAME	DIFF
B	0.73	1.1	15.03	1.42	SAME	DIFF
C	11.27	0.81	16.87	1.16	SAME	DIFF
D	8.37	5.09	7.42	3.41	SAME	SAME
POLYTHENE	1.4	1.09	9.72	0.48	SAME	DIFF
ACRYLIC	2.52	0.42	7.78	0.58	SAME	DIFF

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 1.4 (CONT.)

UNCOATED V. IRON ZINC ALLOY - (12 MONTHS)

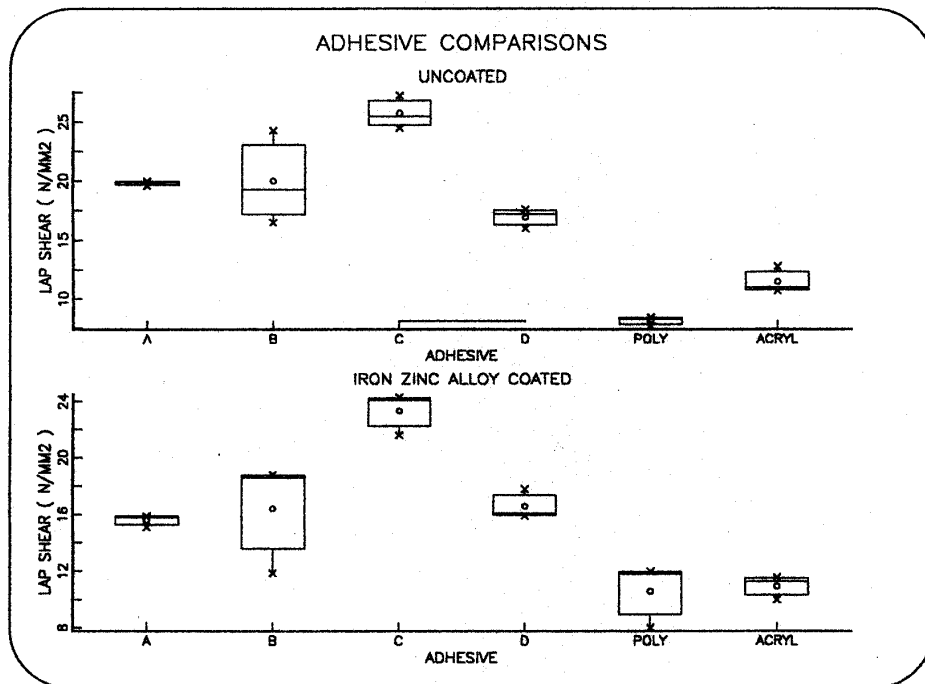
ADHESIVE	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
A	3.37	4.37	9.17	0.58	DIFF	SAME
B	0.13	0.06	14.5	1.05	DIFF	DIFF
C	7.85	0.84	13.9	1.54	SAME	DIFF
D	5.47	1.28	7.67	3.89	SAME	SAME
POLYTHENE	0.13	0.06	7.58	2.12	DIFF	DIFF
ACRYLIC	0.13	0.06	9.32	1.9	DIFF	DIFF

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.

2. A significance level of 5% was chosen for all comparisons.

APPENDIX 1.5

ADHESIVE COMPARISONS (0 MONTHS)

COATING	ADHESIVE												ADHESIVE COMPARISONS	
	A		B		C		D		POLY.		ACRYL.		BARTLETT SD TEST	ANOVA AVERAGE TEST
	AVE	SD	AVE	SD	AVE	SD	AVE	SD	AVE	SD	AVE	SD		
UNCOATED	19.83	0.21	20.03	3.95	25.77	1.42	16.93	0.83	8.13	0.39	11.5	1.14	DIFF	DIFF
IRON ZINC ALLOY	15.6	0.44	16.43	3.92	23.3	1.5	16.6	1.04	10.6	2.25	10.97	0.85	SAME	DIFF

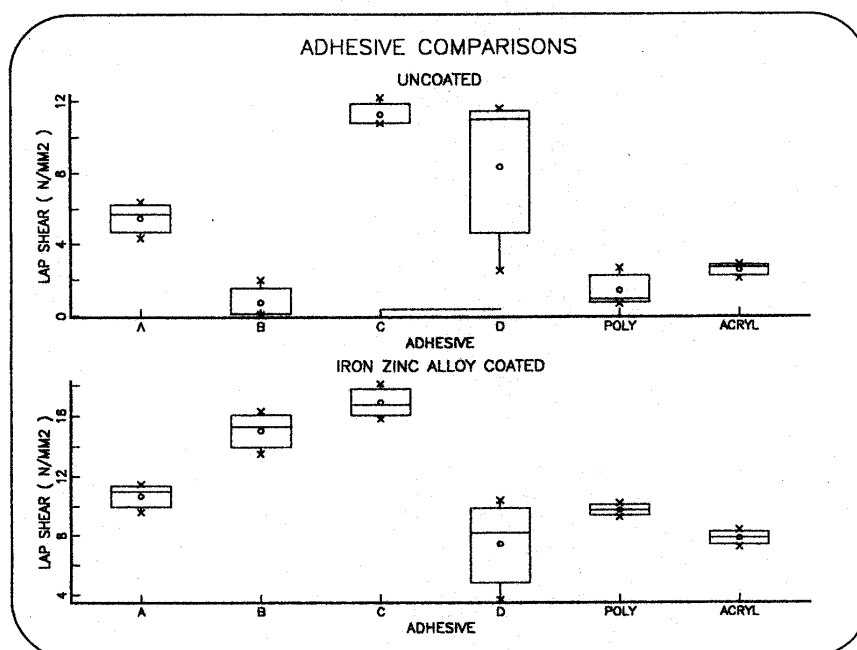
SIGNIFICANCE OF ADHESIVES

COATING	RANKING (LOWEST TO HIGHEST N/MM ²)						
		1	2	3	4	5	6
UNCOATED	ADHESIVE	POLY	ACRYL	D	A	B	C

IRON ZINC ALLOY	ADHESIVE	POLY	ACRYL	A	B	D	C

NOTE. THE ADHESIVES Banded BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBanded ADHESIVES ARE DIFFERENT FROM EACH OTHER

APPENDIX 1.5 (CONT.)

ADHESIVE COMPARISONS (3 MONTHS)

COATING	ADHESIVE												ADHESIVE COMPARISONS	
	A		B		C		D		POLY.		ACRYL.		BARTLETT SD TEST	ANOVA AVERAGE TEST
	AVE	SD	AVE	SD	AVE	SD	AVE	SD	AVE	SD	AVE	SD		
UNCOATED	5.48	1.04	0.73	1.1	11.27	0.81	8.37	5.09	1.4	1.09	2.52	0.42	DIFF	DIFF
IRON ZINC ALLOY	10.7	0.98	15	1.42	16.87	1.16	7.42	3.41	9.72	0.48	7.78	0.58	SAME	DIFF

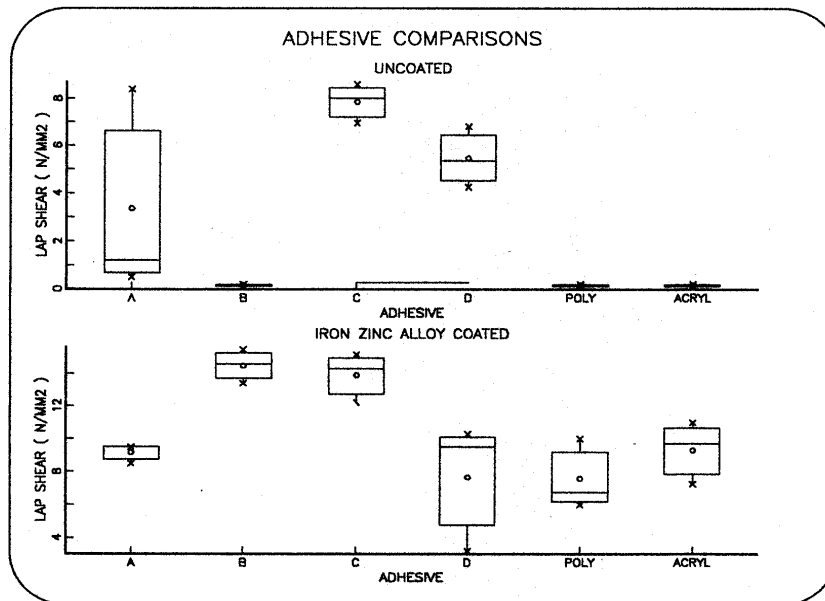
SIGNIFICANCE OF ADHESIVES

COATING	RANKING (LOWEST TO HIGHEST N/MM ²)						
		1	2	3	4	5	6
UNCOATED	ADHESIVE	B	POLY	ACRYL	A	D	C

IRON ZINC ALLOY	ADHESIVE	D	ACRYL	POLY	A	B	C

NOTE. THE ADHESIVES BANNED BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBANNED ADHESIVES ARE DIFFERENT FROM EACH OTHER

APPENDIX 1.5 (CONT.)

ADHESIVE COMPARISONS (12 MONTHS)

COATING	ADHESIVE												ADHESIVE COMPARISONS	
	A	SD	B	SD	C	SD	D	SD	POLY	SD	ACRYL	SD	BARTLETT SD TEST	ANOVA AVERAGE TEST
UNCOATED	3.37	4.37	0.13	0.06	7.85	0.84	5.47	1.28	0.13	0.06	0.13	0.06	DIFF	DIFF
IRON ZINC ALLOY	9.17	0.58	14.5	1.05	13.9	1.54	7.67	3.89	7.58	2.12	9.32	1.9	SAME	DIFF

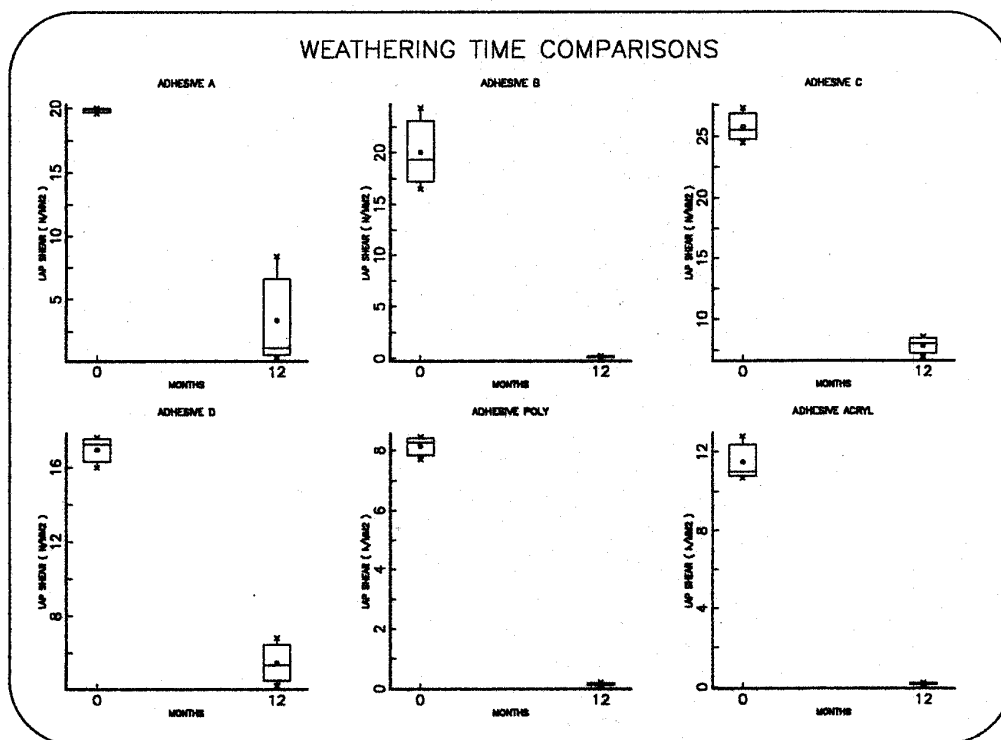
SIGNIFICANCE OF ADHESIVES

		RANKING (LOWEST TO HIGHEST N/MM ²)					
COATING		1	2	3	4	5	6
UNCOATED	ADHESIVE	B	POLY	ACRYL	A	D	C

IRON ZINC ALLOY	ADHESIVE	POLY	D	A	ACRYL	C	B

NOTE. THE ADHESIVES BANDED BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBANDED ADHESIVES ARE DIFFERENT FROM EACH OTHER

APPENDIX 1.6

WEATHERING TIME COMPARISONS (UNCOATED STEEL)

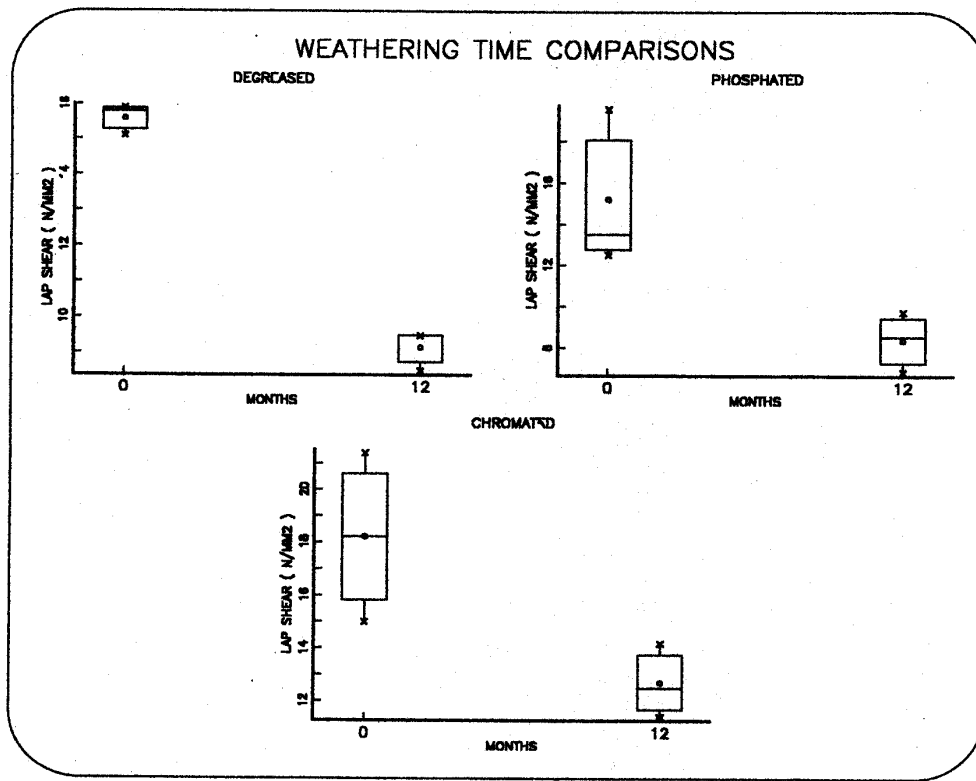
ADHESIVE	TIME 0 MONTHS		TIME 12 MONTHS		TIME COMPARISONS		
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE TEST	% REDUCTION 0 TO 12 MONTHS
A	19.83	0.21	3.37	4.37	DIFF	DIFF	83
B	20.03	3.95	0.13	0.06	DIFF	DIFF	100
C	25.77	1.42	7.85	0.84	SAME	DIFF	70
D	16.93	0.83	5.47	1.28	SAME	DIFF	68
POLYTHENE	8.13	0.39	0.13	0.06	DIFF	DIFF	98
ACRYLIC	11.5	1.14	0.13	0.06	DIFF	DIFF	99

AVERAGE REDUCTION = 86%

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 1.6 (CONT.)

WEATHERING TIME COMPARISONS (IRON ZINC ALLOY COATED STEEL)

SURFACE TREATMENT	TIME 0 MONTHS		TIME 12 MONTHS		TIME COMPARISONS		
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE TEST	% REDUCTION 0 TO 12 MONTHS
DEGREASED	15.6	0.44	9.17	0.58	SAME	DIFF	41
PHOSPHATED	15.2	3.84	8.38	1.46	SAME	DIFF	45
CHROMATED	18.2	3.2	12.7	1.41	SAME	SAME	30

AVERAGE REDUCTION = 39%

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.

2. A significance level of 5% was chosen for all comparisons.

APPENDIX 2.1

ORIGINAL DATA FROM BRITISH STEEL (REF. REPORT NO. EUR 17998 EN)

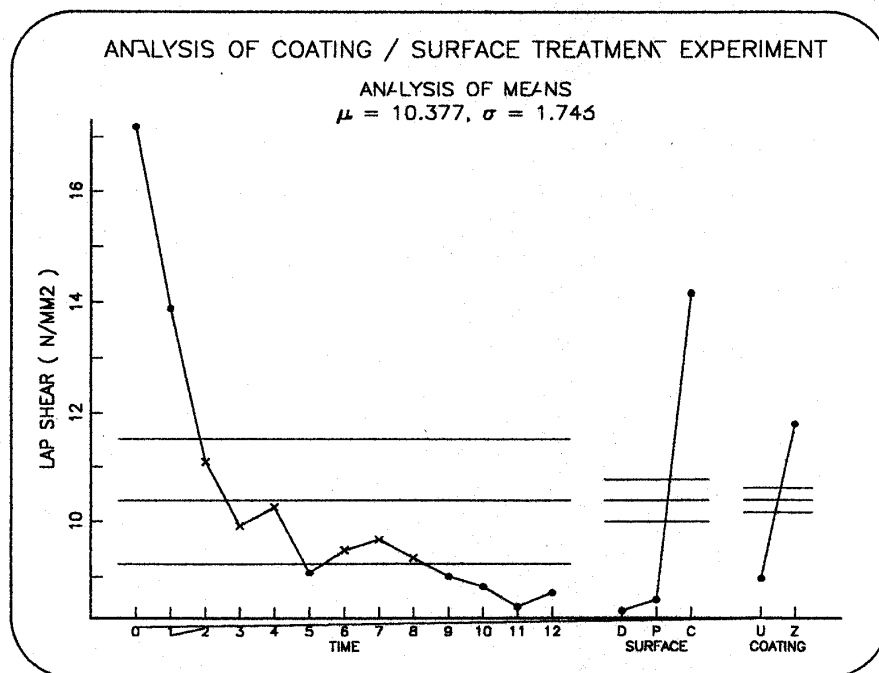
Surface	1.22mm uncoated MILD STEEL- (Adhesive A)												
	Exposure time (months)												
	0	1	2	3	4	5	6	7	8	9	10	11	12
Degreased	19.6	22.8	7.35	4.35	5.3	3.25	2.95	5.35	3.1	3.75	0.1	2.25	8.4
	20	22.8	10.4	5.7	4.1	6.7	0.1	4.35	7.9	1.1	0.2	3.05	1.2
Phosphated	19.9	11.3	7.35	6.4	3.4	0.85	3	4.65	2.9	4.2	0.1	2.5	0.5
	11.3	10.1	7.75	6.35	6.7	5.85	8	7.4	4.9	5.8	4.85	4.4	5.85
	13.3	8.65	8.7	6.6	6.65	5.8	6	5	4.7	6.25	6.65	5	4.89
Chromated	9.35	8.1	6.1	5.8	4.25	5.9	7	6.95	5	5.3	4.9	6.05	4.7
	23.3	15.5	11.9	13.5	13.3	12.5	12.2	13.7	10.4	13.3	12.9	10.9	14.6
	26	15.9	12.4	13.7	16.9	12.7	12.2	12	13.5	12.9	12.4	11.2	15.1
	19.5	15	18.3	12	13.1	13	13.2	12.8	17.4	13.8	13.4	11.5	10.8

Surface	1.1mm IRON -ZINC ALLOY coated steel (Adhesive A)												
	Exposure time (months)												
	0	1	2	3	4	5	6	7	8	9	10	11	12
Degreased	15.8	11.4	10.3	11.5	9.25	9.65	8.4	10.2	9.15	8.95	14.8	10.1	8.5
	15.9	12.1	9.35	9.6	9.7	9.1	11.2	10.4	8.35	9.65	11.5	9.5	9.5
	15.1	8.7	10.7	11	9.9	11.2	9.85	9.4	10.8	9.7	10.5	10.3	9.5
Phosphated	12.5	13	11.8	11.7	11.8	10.4	10	9.35	10.7	8.7	8.05	9.6	8.55
	13.5	12.5	12.2	10.9	12.1	8.6	10.5	9.65	10.7	9.65	9.45	9.5	6.85
	19.6	13.7	9.9	10.9	11.3	9.5	10.3	11.5	9.35	7.65	8.1	8.75	9.75
Chromated	18.2	13.7	15.9	11.4	15.7	10.8	16.2	12.5	14	13	14.3	9.6	12.5
	15	16.2	14.1	11.8	15.5	13.4	15.2	15.5	13.9	14.6	14.4	15.9	14.2
	21.4	18.7	15.1	15.3	15.7	14.1	14.3	13.3	11.4	13.8	12.2	12.1	11.4

APPENDIX 2.2

SURFACE TREATMENT / COATING / TIME EXPERIMENTANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F RATIO	SIG. LEVEL
T TIME	12	1336.8	111.4	36.6	3.3E-16
S SURFACE	2	1682.4	841.2	276.2	2.2E-16
C COATING	1	467.1	467.1	153.4	0
TS	24	215.2	9	2.9	3.0E-5
TC	12	228.2	19	6.2	6.2E-9
SC	2	203.3	101.7	33.4	8.5E-13
TSC	24	341.4	14.2	4.7	1.4E-9
ERROR (PURE)	156	475.1	3.05		
TOTAL	233	4949.4	21.24		

ANALYSIS OF MEANS - MAIN EFFECTS

NOTES.

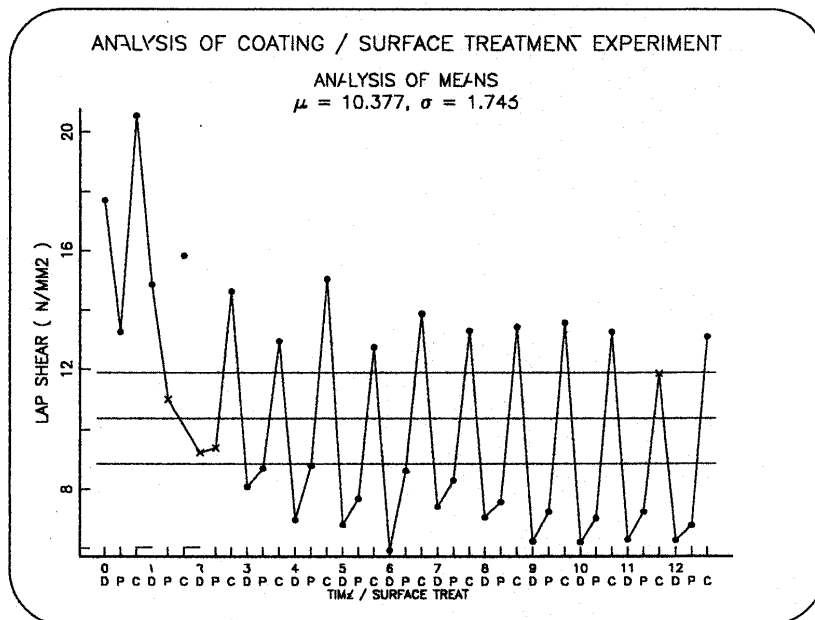
Surface D = Degreased; Surface P = Phosphated; Surface C = Chromated
 Coating U = Uncoated ; Coating Z = Iron Zinc Alloy

APPENDIX 2.3

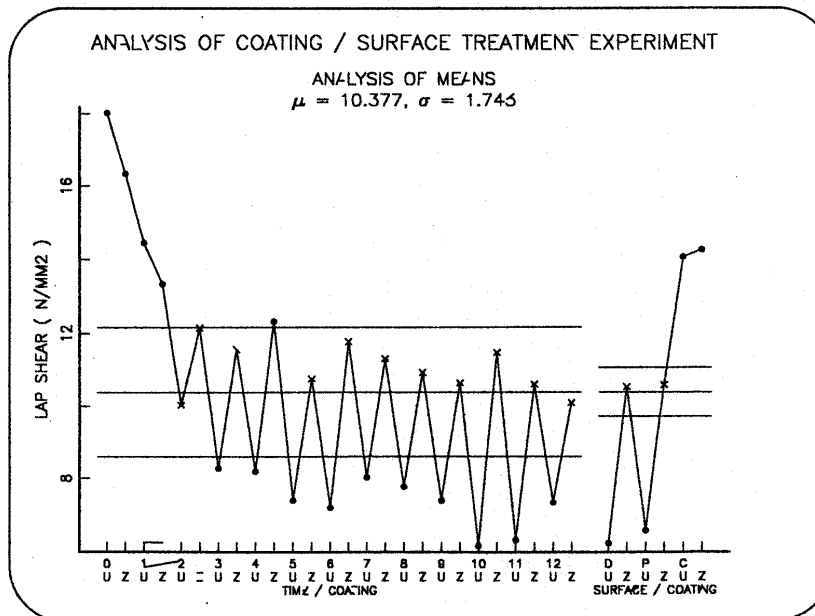
APPENDIX 2.3

ANALYSIS OF MEANS - INTERACTION EFFECTS

TIME / SURFACE (TS)



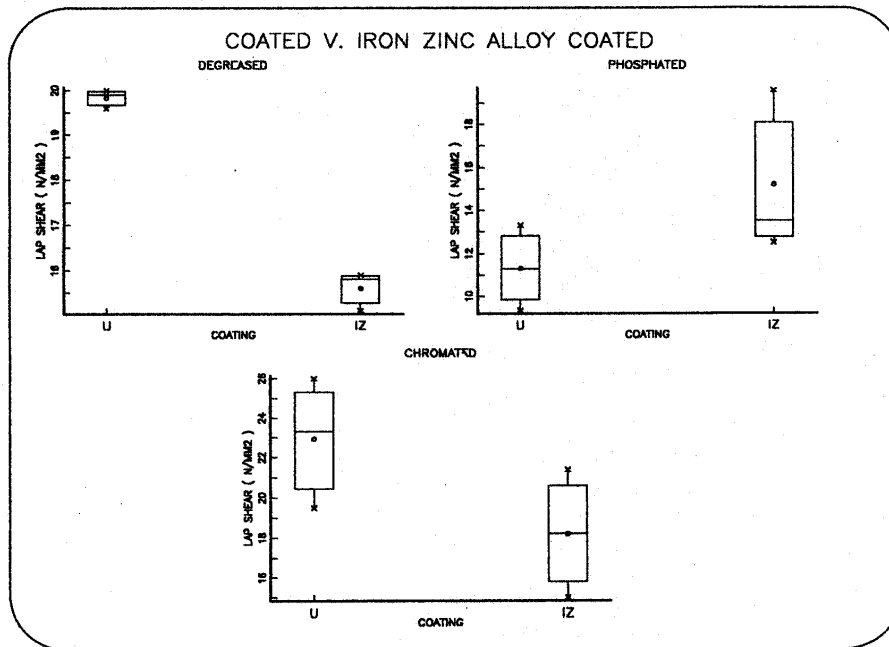
TIME / COATING (TC) ----- SURFACE / COATING (SC)



NOTES.

Surface D = Degreased; Surface P = Phosphated; Surface C = Chromated
 Coating U = Uncoated ; Coating Z = Iron Zinc Alloy

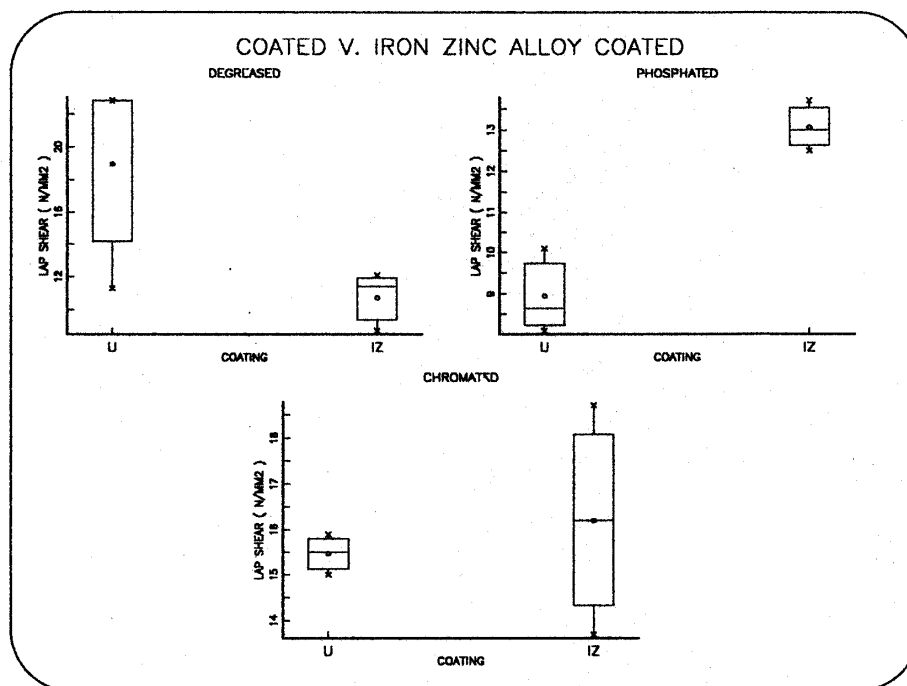
APPENDIX 2.4

UNCOATED V. IRON ZINC ALLOY - (0 MONTHS)

SURFACE TREATMENT	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
DEGREASED	19.83	0.21	15.6	0.44	SAME	DIFF
PHOSPHATED	11.32	1.98	15.2	3.84	SAME	SAME
CHROMATED	22.93	3.27	18.2	3.2	SAME	SAME

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

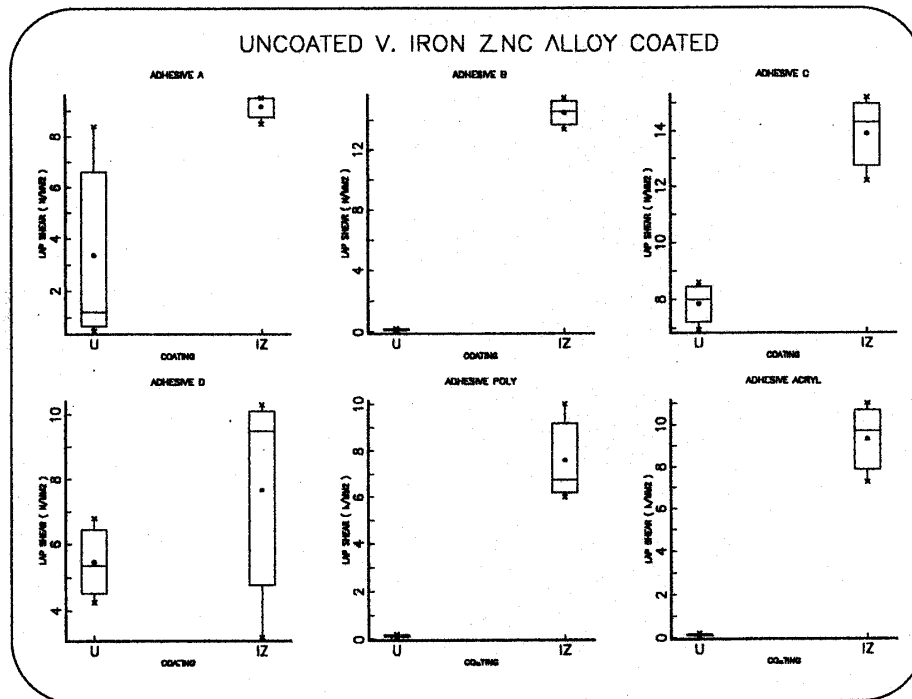
APPENDIX 2.4 (CONT.)**UNCOATED V. IRON ZINC ALLOY - (3 MONTHS)**

SURFACE TREATMENT	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
DEGREASED	18.97	6.64	10.93	1.8	SAME	SAME
PHOSPHATED	8.95	1.03	13.07	0.6	SAME	DIFF
CHROMATED	15.47	0.45	16.2	2.5	SAME	SAME

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 2.4 (CONT.)

UNCOATED V. IRON ZINC ALLOY - (12 MONTHS)

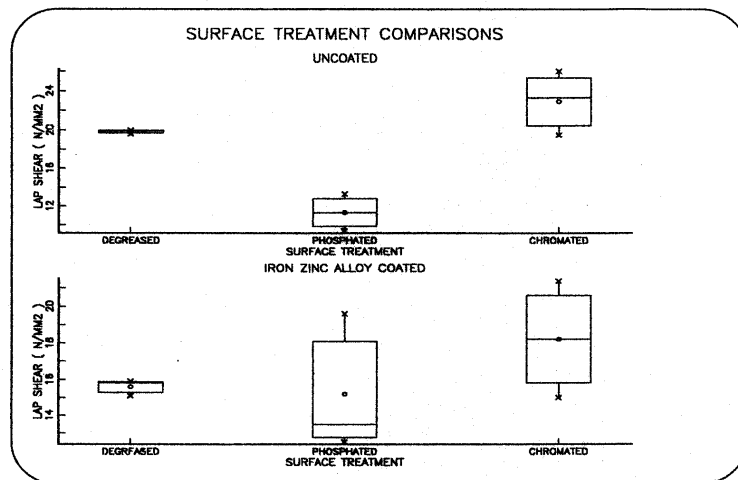
ADHESIVE	UNCOATED		IRON ZINC ALLOY		U v. IZ COMPARISONS	
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE
A	3.37	4.37	9.17	0.58	DIFF	SAME
B	0.13	0.06	14.5	1.05	DIFF	DIFF
C	7.85	0.84	13.9	1.54	SAME	DIFF
D	5.47	1.28	7.67	3.89	SAME	SAME
POLYTHENE	0.13	0.06	7.58	2.12	DIFF	DIFF
ACRYLIC	0.13	0.06	9.32	1.9	DIFF	DIFF

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 2.5

APPENDIX 2.5

SURFACE TREATMENT COMPARISONS (0 MONTHS)

COATING	SURFACE TREATMENT						SURFACE TREATMENT COMPARISONS	
	DEGREASED	PHOSPHATED	CHROMATED	AVE.	SD	AVE.	BARTLETT SD TEST	ANOVA AVERAGE TEST
UNCOATED	19.83	0.21	11.32	1.98	22.93	3.27	DIFF	DIFF
IRON ZINC ALLOY	15.6	0.44	15.2	3.84	18.2	3.2	SAME	SAME

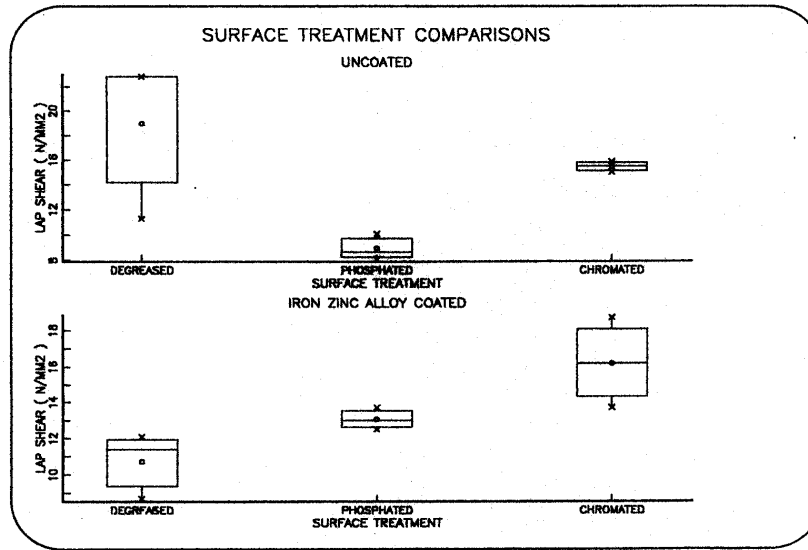
SIGNIFICANCE OF SURFACE TREATMENTS

COATING	RANKING (LOWEST TO HIGHEST N/MM ²)			
		1	2	3
UNCOATED	SURFACE	PHOSPHATED	DEGREASED	CHROMATED

IRON ZINC ALLOY	SURFACE	PHOSPHATED	DEGREASED	CHROMATED

NOTE. THE SURFACE TREATMENTS Banded BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBanded SURFACE TREATMENTS ARE DIFFERENT FROM EACH OTHER

APPENDIX 2.5 (CONT.)

SURFACE TREATMENT COMPARISONS (3 MONTHS)

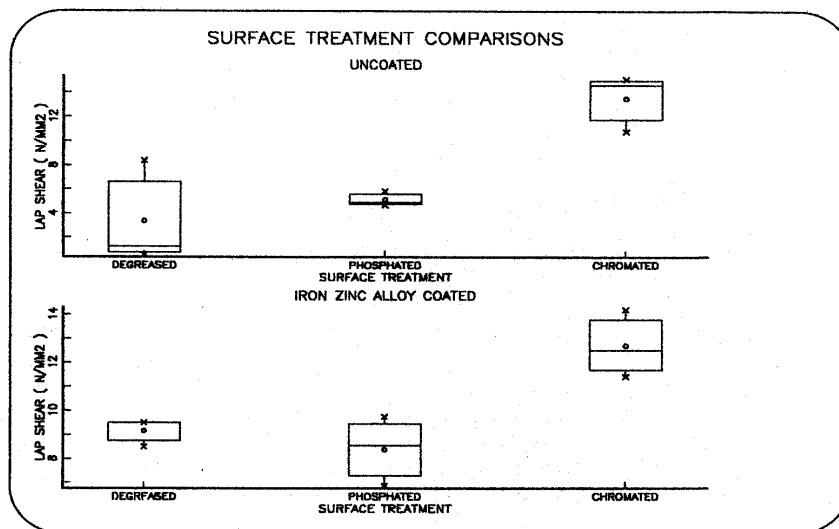
COATING	SURFACE TREATMENT						SURFACE TREATMENT COMPARISONS	
	DEGREASED		PHOSPHATED		CHROMATED		BARTLETT SD TEST	ANOVA AVERAGE TEST
	AVE.	SD	AVE.	SD	AVE.	SD		
UNCOATED	18.97	6.64	8.95	1.03	15.47	0.45	DIFF	DIFF
IRON ZINC ALLOY	10.73	1.8	13.07	0.6	16.2	2.5	SAME	DIFF

SIGNIFICANCE OF SURFACE TREATMENTS

COATING	RANKING (LOWEST TO HIGHEST N/MM ²)			
		1	2	3
UNCOATED	SURFACE	PHOSPHATED	CHROMATED	DEGREASED
		*****	*****	*****
IRON ZINC ALLOY	SURFACE	DEGREASED	PHOSPHATED	CHROMATED
		*****	*****	*****

NOTE. THE SURFACE TREATMENTS Banded BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBanded SURFACE TREATMENTS ARE DIFFERENT FROM EACH OTHER

APPENDIX 2.5 (CONT.)

SURFACE TREATMENT COMPARISONS (12 MONTHS)

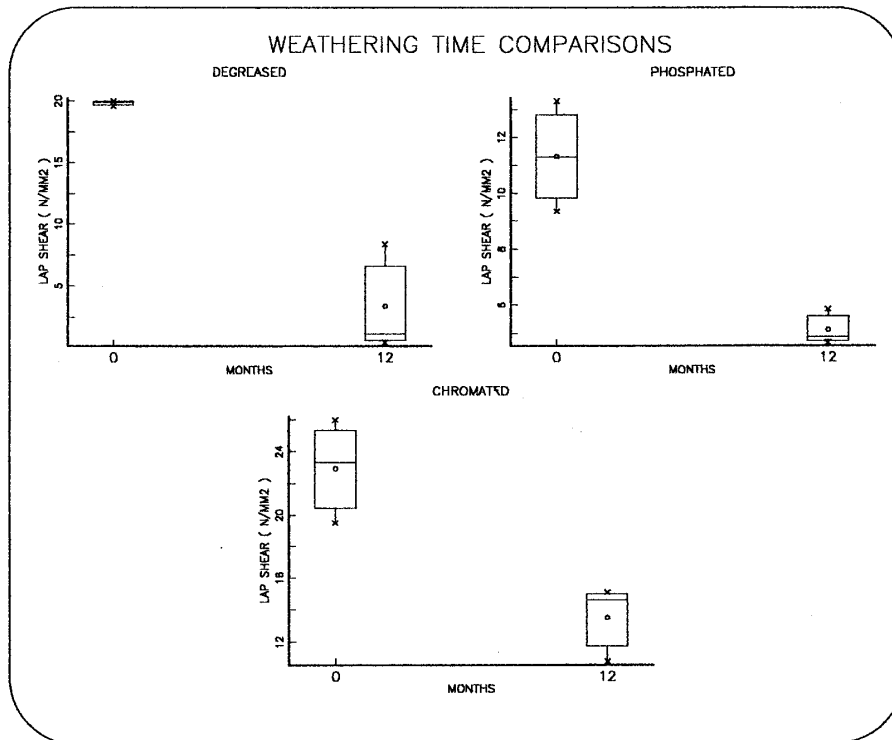
COATING	SURFACE TREATMENT						SURFACE TREATMENT COMPARISONS	
	DEGREASED		PHOSPHATED		CHROMATED		BARTLETT SD TEST	ANOVA AVERAGE TEST
	AVE.	SD	AVE.	SD	AVE.	SD		
UNCOATED	3.37	4.37	5.15	0.62	13.5	2.35	SAME	DIFF
IRON ZINC ALLOY	9.17	0.58	8.38	1.46	12.7	1.41	SAME	DIFF

SIGNIFICANCE OF SURFACE TREATMENTS				
RANKING (LOWEST TO HIGHEST N/MM ²)				
COATING		1	2	3
UNCOATED	SURFACE	DEGREASED	PHOSPHATED	CHROMATED

IRON ZINC ALLOY	SURFACE	PHOSPHATED	DEGREASED	CHROMATED

NOTE. THE SURFACE TREATMENTS Banded BY THE ***** INDICATES THEY ARE NOT SIGNIFICANTLY DIFFERENT. ALL UNBanded SURFACE TREATMENTS ARE DIFFERENT FROM EACH OTHER

APPENDIX 2.6

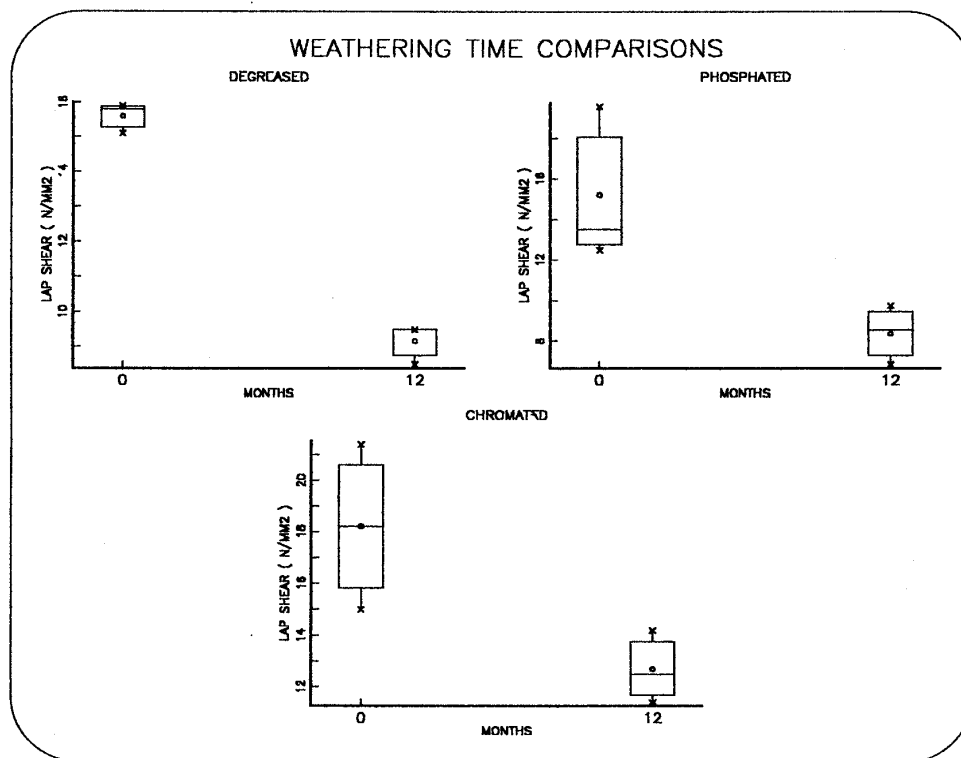
WEATHERING TIME COMPARISONS (UNCOATED STEEL)

SURFACE TREATMENT	TIME 0 MONTHS		TIME 12 MONTHS		TIME COMPARISONS		
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE TEST	% REDUCTION 0 TO 12 MONTHS
DEGREASED	19.83	0.21	3.37	4.37	DIFF	DIFF	83
PHOSPHATED	11.32	1.98	5.15	0.62	SAME	DIFF	55
CHROMATED	22.93	3.27	13.5	2.35	SAME	DIFF	41

AVERAGE REDUCTION = 60%

NOTES.

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

APPENDIX 2.6 (CONT.)**WEATHERING TIME COMPARISONS (IRON ZINC ALLOY COATED STEEL)**

SURFACE TREATMENT	TIME 0 MONTHS		TIME 12 MONTHS		TIME COMPARISONS		
	AVERAGE	S.DEV	AVERAGE	S.DEV	BARTLETT SD TEST	ANOVA AVERAGE TEST	% REDUCTION 0 TO 12 MONTHS
DEGREASED	15.6	0.44	9.17	0.58	SAME	DIFF	41
PHOSPHATED	15.2	3.84	8.38	1.46	SAME	DIFF	45
CHROMATED	18.2	3.2	12.7	1.41	SAME	SAME	30

AVERAGE REDUCTION = 39%**NOTES.**

1. BARTLETT SD TEST. - SAME means the comparison of either S.DEV's or AVERAGE'S are not significantly different.
- DIFF means the comparison is significantly different.
2. A significance level of 5% was chosen for all comparisons.

Case Study 2
Statistical Comparisons of
Lap-Shear and Thick-Adherend Shear Test Data

1. INTRODUCTION

This cases study involves the examination of data produced by the American Cyanamid Company on adhesives FXM 350-NA and FXM 350U-NA under various environmental conditions and test regimes [1]. The analyses in this case study focuses on the results obtained for FM 350-NA from lap shear and thick adherend shear tests conducted under different temperature conditions. The case study compares the two test methods for statistical significance. The data was supplied to the programme courtesy of British Aerospace, Sowerby.

2. INITIAL DATA ANALYSIS

Tests were conducted on three different batches of FM 350NA adhesive (D, E and F) under different temperature conditions (-67 °F (-55 °C) to 350 °F (177 °C)) using lap shear and thick adherend shear test (TAST) methods. Ten lap shear and five thick adherend specimens from each batch were tested at each condition. The data (i.e. ultimate shear strength in psi) examined in this report is contained in Appendix 1.1.

2.1 LAP SHEAR VERSUS THICK ADHEREND COMPARISONS

As there was sufficient data available, it was possible to compare the lap shear results against TAST results over a wide range of temperatures. An initial view of the results is presented as Box plots in Appendix 1.2. These plots show the relationship between lap shear and thick adherend results for each temperature and batch of adhesive. The most obvious result is that the thick adherend results are much larger than the lap shear results for all test conditions.

A more formal analysis of the data is shown in Appendix 1.3 in which averages and standard deviations of each group of results are presented. The table in Appendix 1.3 also contains the formal tests of statistical significance for the comparison of the averages and standard deviations. These tests are referred to as the Bartlett Standard Deviation test and ANOVA Average test. It can be seen that the ANOVA Average test concludes that all the comparisons are different (i.e. lap shear is much smaller than thick adherend over all test conditions).. This conclusion agrees with the results observed from the Box plots. However, in addition, the Bartlett Standard Deviation test shows that for:

-67 °F Standard deviation of the lap shear and thick adherend test data are the same.

75 °F Standard deviation of the lap shear test is smaller than for TAST.

350 °F Standard deviation shows inconsistency between batches with no obvious pattern.

These results may seem surprising when viewed against the magnitude of the standard deviation being compared (e.g. 109.3 psi for lap shear against 196.8 psi for TAST (-67 °F/Batch D)), which is not considered from a statistical perspective to be significantly different. This highlights one of the problems of understanding whether standard deviations are different when the sample sizes are relatively small. One way of solving the small sample size problem is by grouping the results that are essentially part of the same

homogeneous group. The three batches used in the study could form a homogeneous group if there were no batch to batch differences between each combination of D, E and F.

3. BATCH COMPARISONS

The batch to batch comparisons for the two test methods are shown in Appendices 2.1 and 2.2 (see Box plots). The table under the Box plots shows the batch details in terms of averages and standard deviations. In addition, the table includes the formal Bartlett test for standard deviations and ANOVA test for averages.

3.1 LAP SHEAR - BATCH COMPARISONS

The results, presented in Appendix 2.1, show that for:

- 67 °F The batches have the same average and standard deviation.
- 75 °F The batches have the same standard deviation, but different averages.
- 350 °F The batches have the same average and standard deviation.

This means that for -67 °F and 350 °F, the batches are behaving essentially the same. In these circumstances it is reasonable to group the batch results together in a sample of 30 results for the test conditions. For the 75 °F, however, the fact that the batch averages are different means that the batch results cannot be merged into one group and treated in the same manner as the results for the other two temperatures. It is perhaps worth trying to understand why the results should be so different from each other and inconsistent with the findings at -67 °F and 350 °F.

3.2 THICK ADHEREND - BATCH COMPARISONS

The results, presented in Appendix 2.2, show that for:

- 67 °F The batches have the same average and standard deviation.
- 75 °F The batches have the same average and standard deviation.
- 350 °F The batches have the same average and standard deviation.

This means that for all three test temperatures, the batches are behaving essentially the same. It is therefore reasonable to group the batch results together in a sample of 30 results for the test conditions. It can be seen that the inconsistency at 75 °F for the lap shear tests has not carried through to the TAST.

4. COMBINED BATCH ANALYSES

The results of combining the batch results for both lap shear and thick adherend tests are shown in Appendix 3.1 (see Box plot). The table below the Box plot in Appendix 3.1 contains the formal analyses for -67 °F and 350 °F only. The results for 75 °F are excluded due to the differences in batch results observed for the lap shear test. It should be noted that for -67 °F and 350 °F, the averages and standard deviations for the two test methods are different. It should also be noted that although the formal test has not been performed for the 75 °F test results, the comparison of averages and standard deviations is at least of

the same order as that for -67 °F and 350 °F. Therefore, the 75 °F effect is also significant even though the data includes the additional variation caused by differences between batches.

4.1 DISTRIBUTION ANALYSIS

When sample sizes for comparison studies become larger, it is useful understand the distribution form of the results. Previous results have shown that it is possible to combine the results of the batches into homogeneous groups of 30 results.

Appendices 3.2 and 3.3 contain the distribution analysis plots for testing the hypothesis that a set of results come from a Normal distribution. The graphs show the 30 individual results plotted against a Normal distribution scale. If the results plot as a reasonably straight line on this plot then the hypothesis of Normality of the results is reasonable. The results of the analyses presented in Appendices 3.2 and 3.3 are for all sets of data with the exception of the 75 °F lap shear data (for reasons previously discussed). The table included with the results shows the formal “goodness of fit” test referred to as Kolmogorov-Smirnov test. This test shows that results for both test methods, and the test temperatures analysed, follow a Normal distribution characterised by the particular average and standard deviation shown in the table.

5. COMBINED DISTRIBUTIONS

The overall analysis, summarised in Appendix 4.1, shows the calculated distribution of results for the two test methods for each test temperatures -67 °F, 75 °F and 350 °F using the particular averages and standard deviations calculated in Appendices 3.2 and 3.3. To gain a complete picture, the results for 75 °F have been included as a combined batches histogram. The summary table of results is shown in Appendix 4.1 and included below for comment and analysis.

Table 1 Summary of Test Results

Temperature (°F)	Lap Shear (LS)		TAST		LS vs TAST
	Average	S.Dev	Average	S.Dev	Diff. in Average
-67	3890.5	103.8	8457.3	196.4	4566.8
75	4299.7	142.0	9196.5	406.1	4896.8
350	3304.3	121.5	4160.0	282.4	855.7

5.1 RESULTS

The results can be summarised as follows:

- The average result for TAST is significantly higher than for lap shear test. The difference in average strength is 4566.8 psi at -67 °F, 4896.8 psi at 75 °F and 855.7 psi at 350 °F.
- The differences in results is not so marked at 350 °F, although extremely significant.

- The variation in results, measured by the standard deviation, is much higher for the TAST than the lap shear test (196.4 psi versus 103.8 psi) at -67 °F, (406.1 psi versus 142.0 psi) at 75 °F and (282.4 psi versus 121.5 psi) at 350 °F.
- For the batches tested (i.e. D, E and F), there was little batch to batch variation. The only exception was the lap shear data at 75 °F.
- For those cases where it was reasonable to combine batch results into a larger group, the resulting distribution was Normal.

6. CONCLUSIONS

The following conclusions can be drawn from the analyses:

- TAST gave significantly higher average strength results than the lap shear test for all three test temperatures.
- TAST gave significantly higher variation in results than the lap shear test for all three test temperatures.
- The difference noted above means that from a statistical perspective the TAST data is less reliable than the lap shear test over all the temperatures, within the bounds of this study.
- The results imply that since the lap shear test exhibits less variability it is therefore more efficient than the TAST in comparing the effect of material and process parameters on bond strength. One measure of this efficiency is that smaller sample sizes are needed.
- The two previous conclusions can only be strictly applied within the bounds of this experiment. However, a number of interesting thoughts are raised about the TAST (including specimen manufacture) which could benefit from further study.
- The difference in average strengths at 350 °F was markedly reduced compared with the average strengths at -67 °F and 75 °F. The physical cause for this effect is worth further consideration.
- In most cases there was little if no batch effect. The result of this being that the combined set of data from the three batches (D, E and F) could be represented by a Normal distribution.
- The batch effect observed for the lap shear test results at 75 °F is worth further consideration. The batch to batch differences observed for these tests could be due to a real effect or inconsistent experimental practice.
- **It should be noted as a general experimental maxim, that much larger sample sizes are needed to investigate variation effects than are needed to investigate average effects.**

REFERENCES

1. "Qualification Test Report for Rohr Industries, Inc.", American Cyanamid Company, 1987.

ADDITIONAL READING

1. Snedecor and Cochran, "Statistical Methods", Iowa State University Press.
2. Ryan, "Statistical Methods for Quality Improvement, Wiley Interscience.
3. Conover, "Practical Nonparametric Statistics", Wiley.

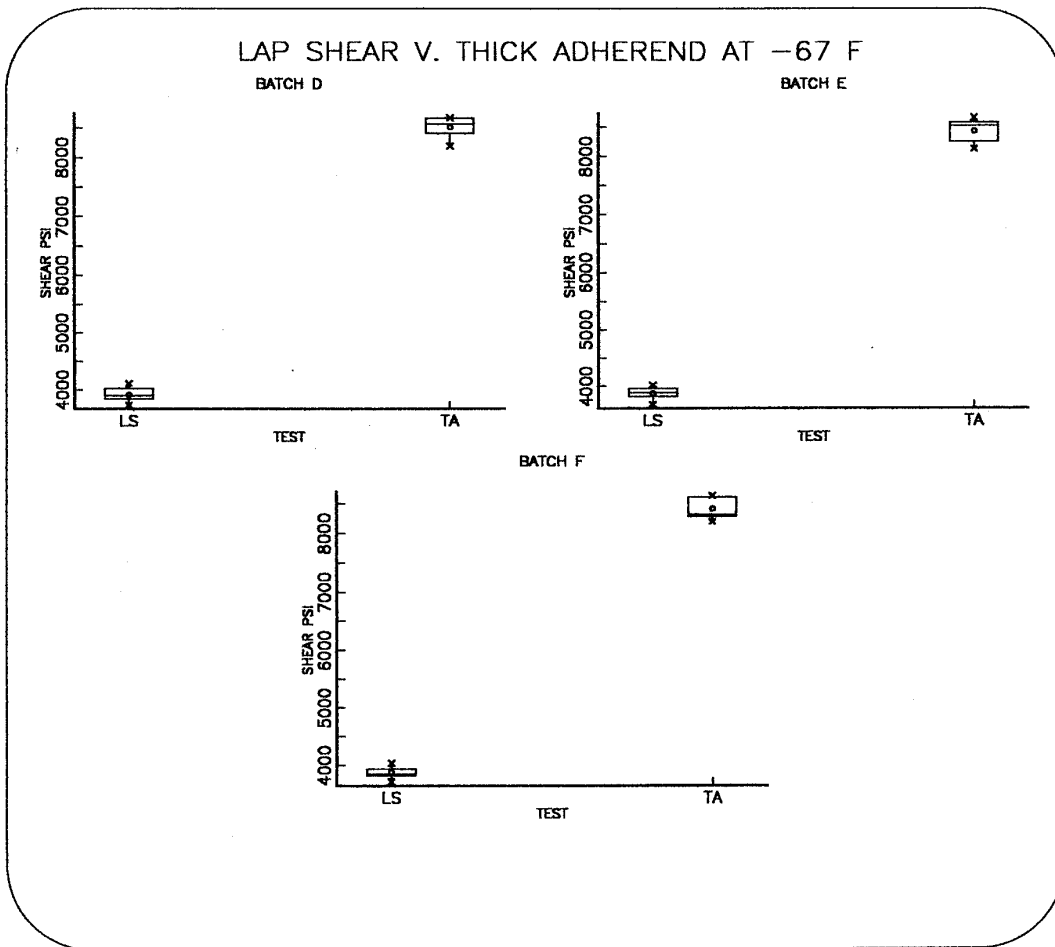
APPENDIX 1.1**THE DATA.**

TEST	TEMPERATURE								
	- 67 F			75 F			350 F		
	BATCH			BATCH			BATCH		
	D	E	F	D	E	F	D	E	F
LAP SHEAR	3834	3990	3912	4324	4154	4268	3382	3106	3297
	3883	3746	3823	4562	4368	4370	3340	3397	3392
	3916	3824	3865	4417	3931	4252	3287	3058	3164
	4025	3919	4010	4573	4125	4461	3474	3397	3327
	3892	3683	3722	4299	4178	4334	3398	3155	3428
	4030	4025	3818	4477	4271	4273	3214	3300	3315
	3741	3911	3930	4325	4077	4153	3427	3397	3316
	4112	3857	3814	4380	4180	4376	3516	3009	3329
	3935	3824	4037	4493	4219	4307	3339	3397	3187
	3844	3966	3828	4336	4212	4296	3315	3203	3263
THICK ADHEREND	8673	8673	8307	9484	8858	9215	4188	4276	4321
	8686	8559	8312	8959	8548	9570	4175	4596	4180
	8488	8130	8604	8539	9547	9373	3843	4429	4451
	8203	8296	8192	9056	9731	8645	4354	3890	4112
	8571	8532	8634	9320	9424	9678	4243	3794	3548

Notes.

1. Data was from the Qualification Test Report . RMS 058, Revision H
2. Adhesive used was FXM 350 - NA
3. Table entries are the ultimate shear psi.

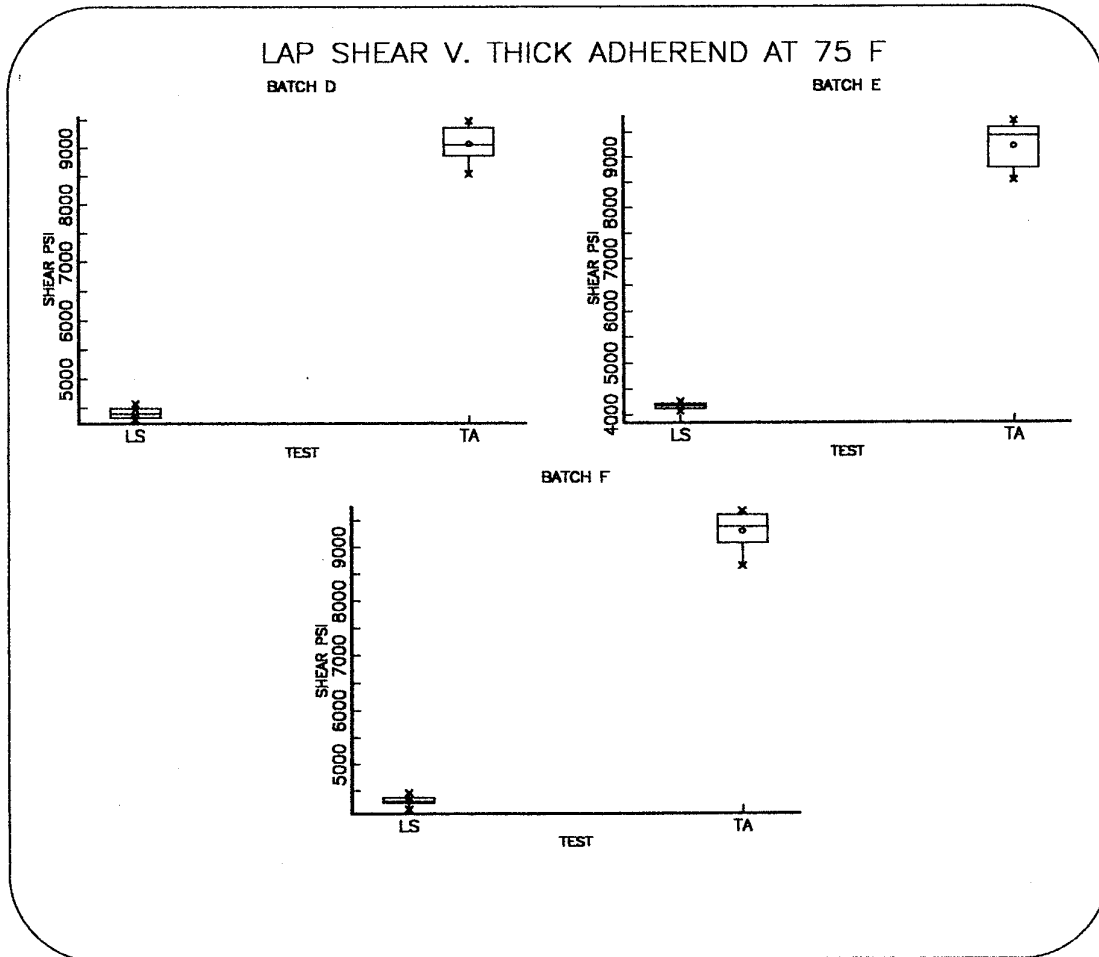
APPENDIX 1.2

LAP SHEAR v. THICK ADHEREND COMPARISONS (-67F)**Note.**

LS = Lap Shear ; TA = Thick Adherend

APPENDIX 1.2 (CONT.)

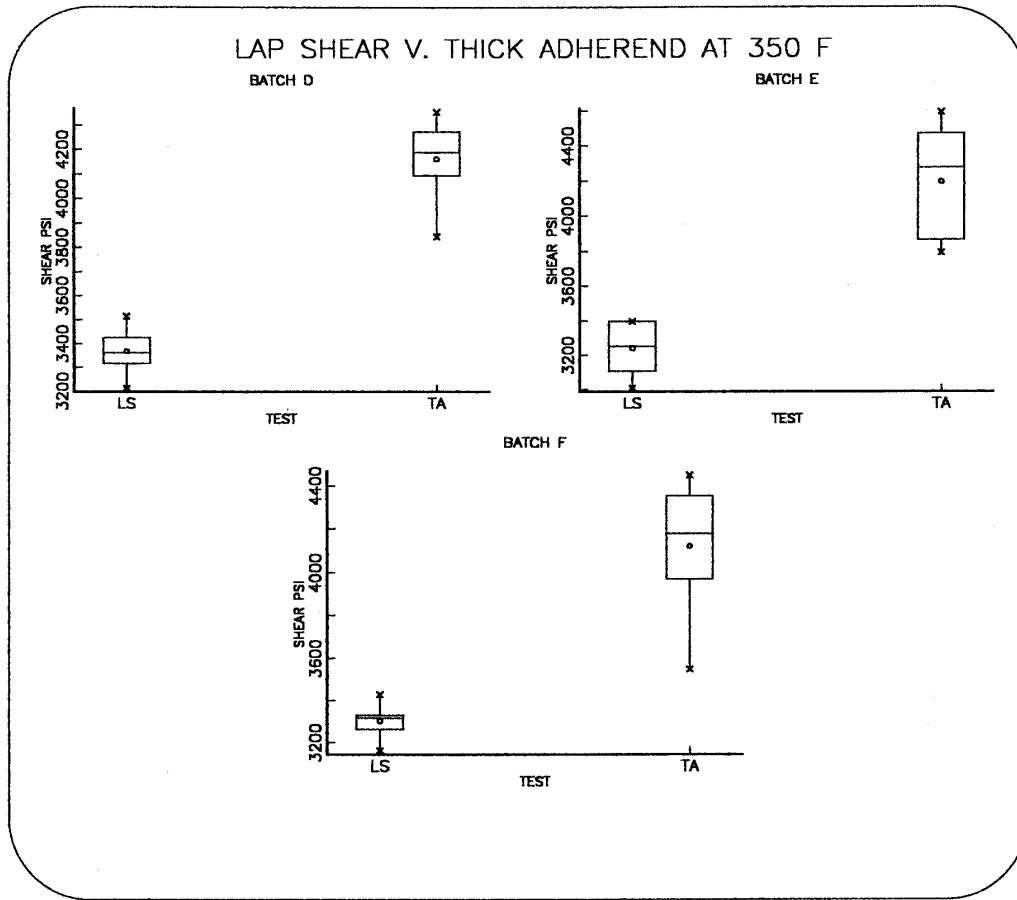
LAP SHEAR v. THICK ADHEREND COMPARISONS (75F)



Note.

LS = Lap Shear ; TA = Thick Adherend

APPENDIX 1.2 (CONT.)

LAP SHEAR v. THICK ADHEREND COMPARISONS (350F)**Note.**

LS = Lap Shear ; TA = Thick Adherend

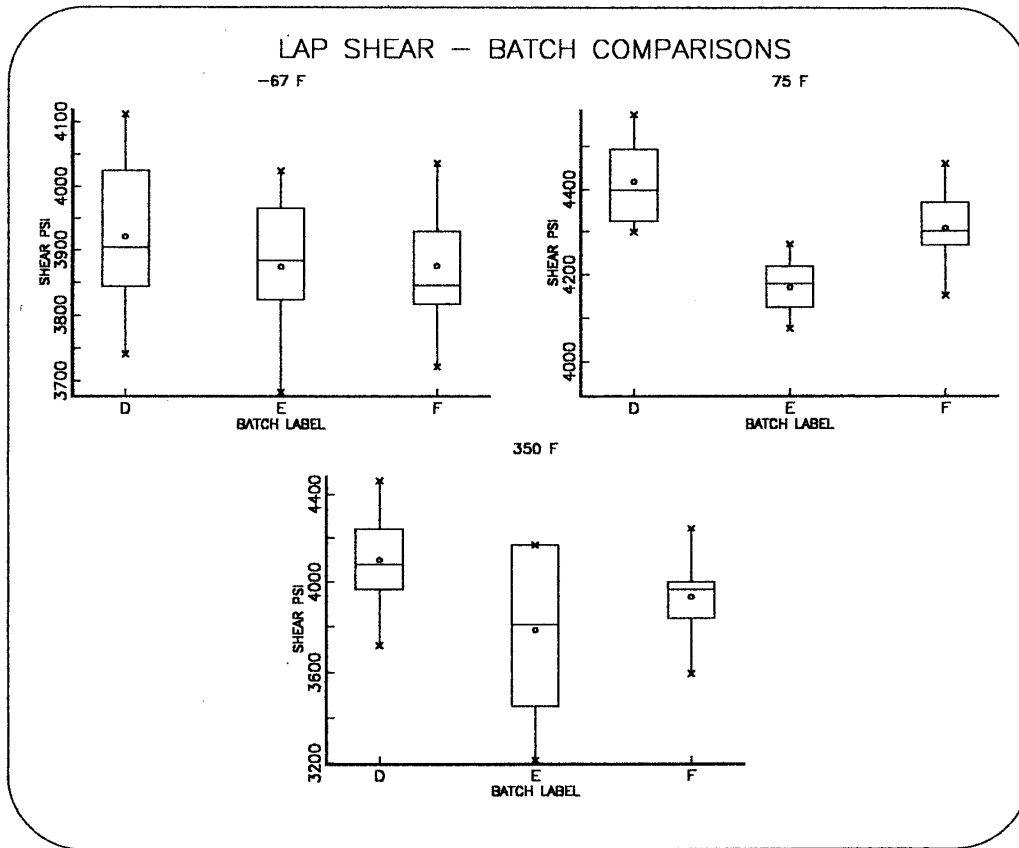
APPENDIX 1.3**LAP SHEAR v. THICK ADHEREND ANALYSIS**

TEMP.	BATCH LABEL	LAP SHEAR		THICK ADHEREND		LS v. TA COMPARISONS	
		AVERAGE	S.DEV.	AVERAGE	S.DEV	BARTLETT S.DEV. TEST	ANOVA AVERAGE TEST
-67 F	D	3921.2	109.3	8524.2	196.8	SAME	DIFF.
	E	3874.5	108.7	8438	220	SAME	DIFF.
	F	3875.9	96.8	8409.8	197.2	SAME	DIFF.
75 F	D	4418.6	102	9071.6	363.4	DIFF.	DIFF.
	E	4175.5	116.3	9221.6	498.1	DIFF.	DIFF.
	F	4309	83.4	9296.2	405.4	DIFF.	DIFF.
350 F	D	3369.2	89.7	4160.6	191.1	SAME	DIFF.
	E	3241.9	154.6	4197	344.9	SAME	DIFF.
	F	3301.8	81.3	4122.4	346.7	DIFF.	DIFF.

Notes.

1. LS = Lap Shear ; TA = Thick Adherend
2. BARTLETT's test for s.dev :
 SAME indicates that the observed difference was not significant.
 DIFF. indicates that the observed difference was significant.
3. ANOVA average test :
 DIFF. indicates that the observed difference was significant.
4. The significance level chosen for all comparisons was 5%

APPENDIX 2.1

LAP SHEAR - BATCH COMPARISONS

TEMP.	BATCH						BATCH COMPARISONS	
	D		E		F		BARTLETT S.DEV TEST	ANOVA AVERAGE TEST
	AVERAGE	S.DEV.	AVERAGE	S.DEV.	AVERAGE	S.DEV.		
-67 F	3921.2	109.3	3874.5	108.7	3875.9	96.8	SAME	SAME
75 F	4418.6	102	4171.5	116.3	4309	83.4	SAME	DIFF.
350 F	3369.2	89.7	3241.9	154.6	3301.8	81.3	SAME	SAME

Notes.

1. BARTLETT's test for s.dev :

SAME indicates that the observed difference was not significant.

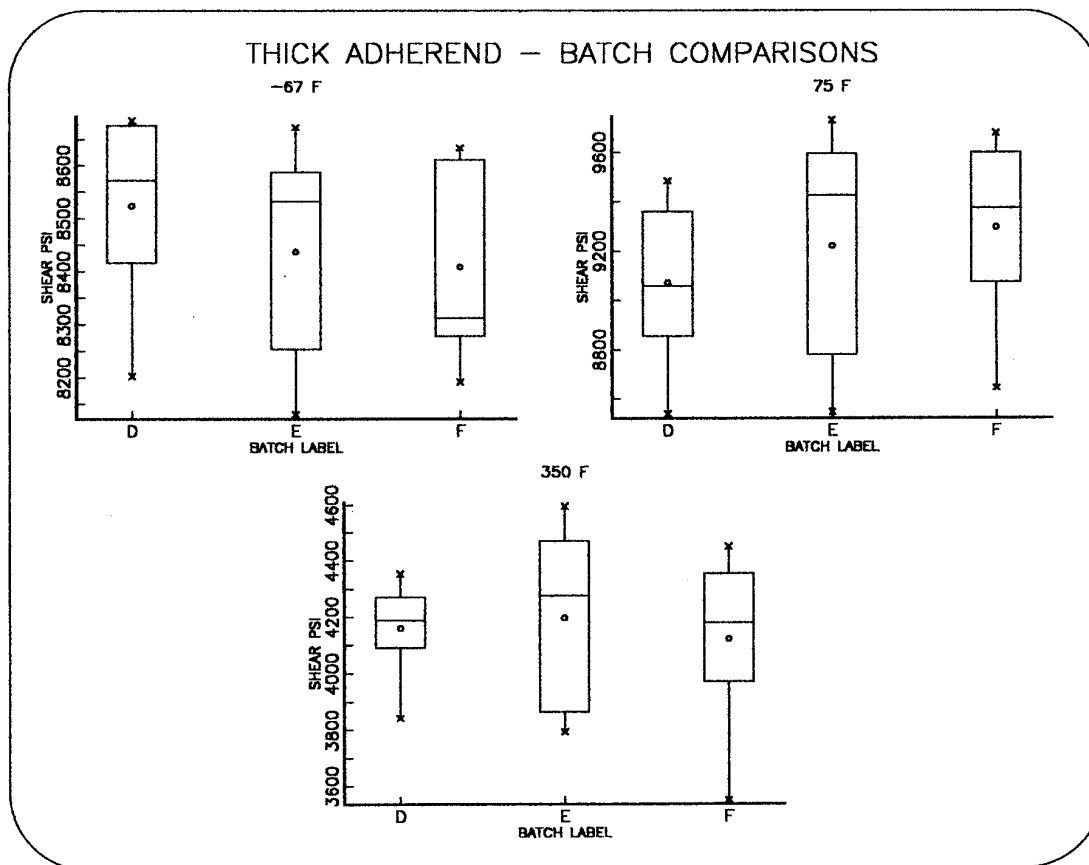
DIFF. indicates that the observed difference was significant.

2. ANOVA average test :

DIFF. indicates that the observed difference was significant.

3. The significance level chosen for all comparisons was 5%

APPENDIX 2.2

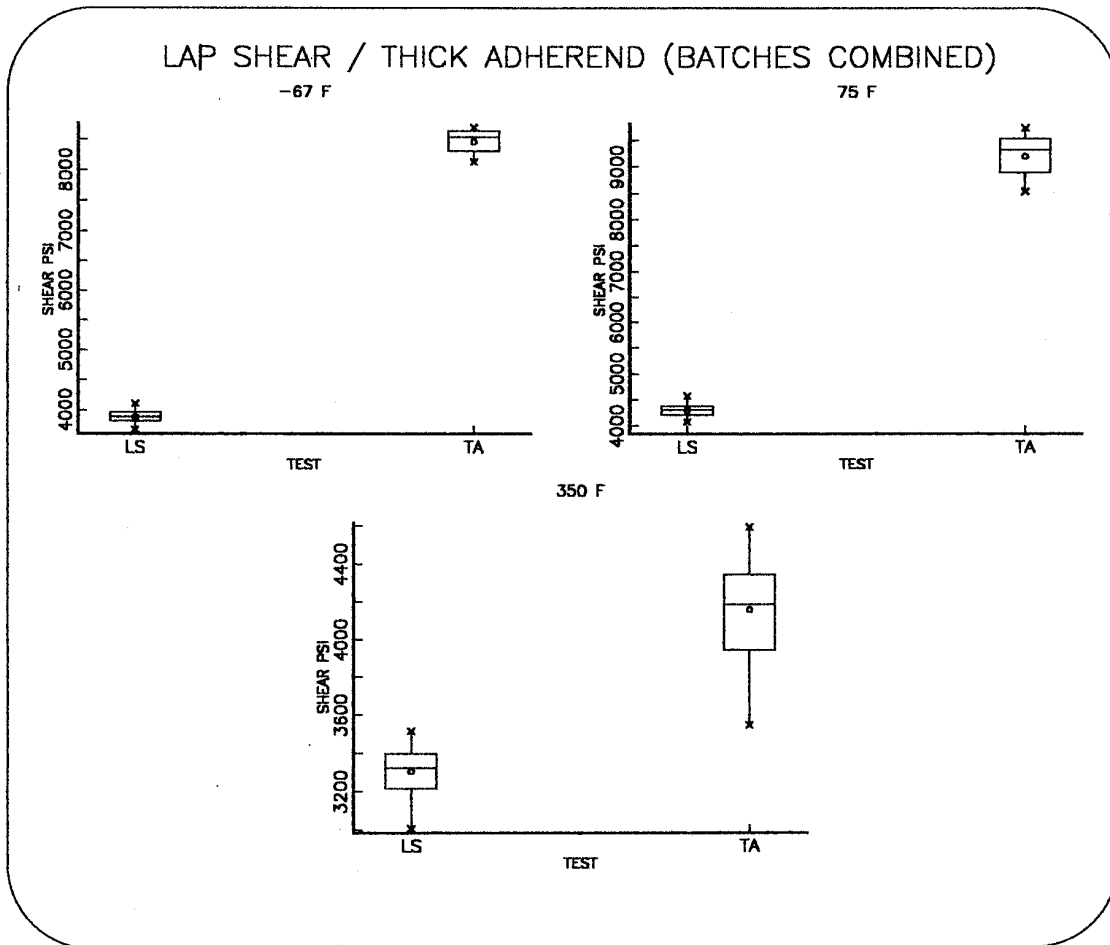
THICK ADHEREND - BATCH COMPARISONS

TEMP.	BATCH						BATCH COMPARISONS	
	D		E		F		BARTLETT S.DEV TEST	ANOVA AVERAGE TEST
	AVERAGE	S.DEV.	AVERAGE	S.DEV.	AVERAGE	S.DEV.		
-67 F	8524.2	196.8	8438	220	8409.8	197.2	SAME	SAME
75 F	9071.6	363.4	9221.6	498	9296.2	405.4	SAME	SAME
350 F	4160.6	191.1	4197	344.9	4122.4	346.7	SAME	SAME

Notes.

1. BARTLETT's test for s.dev :
 SAME indicates that the observed difference was not significant.
 DIFF. indicates that the observed difference was significant.
2. ANOVA average test :
 DIFF. indicates that the observed difference was significant.
3. The significance level chosen for all comparisons was 5%

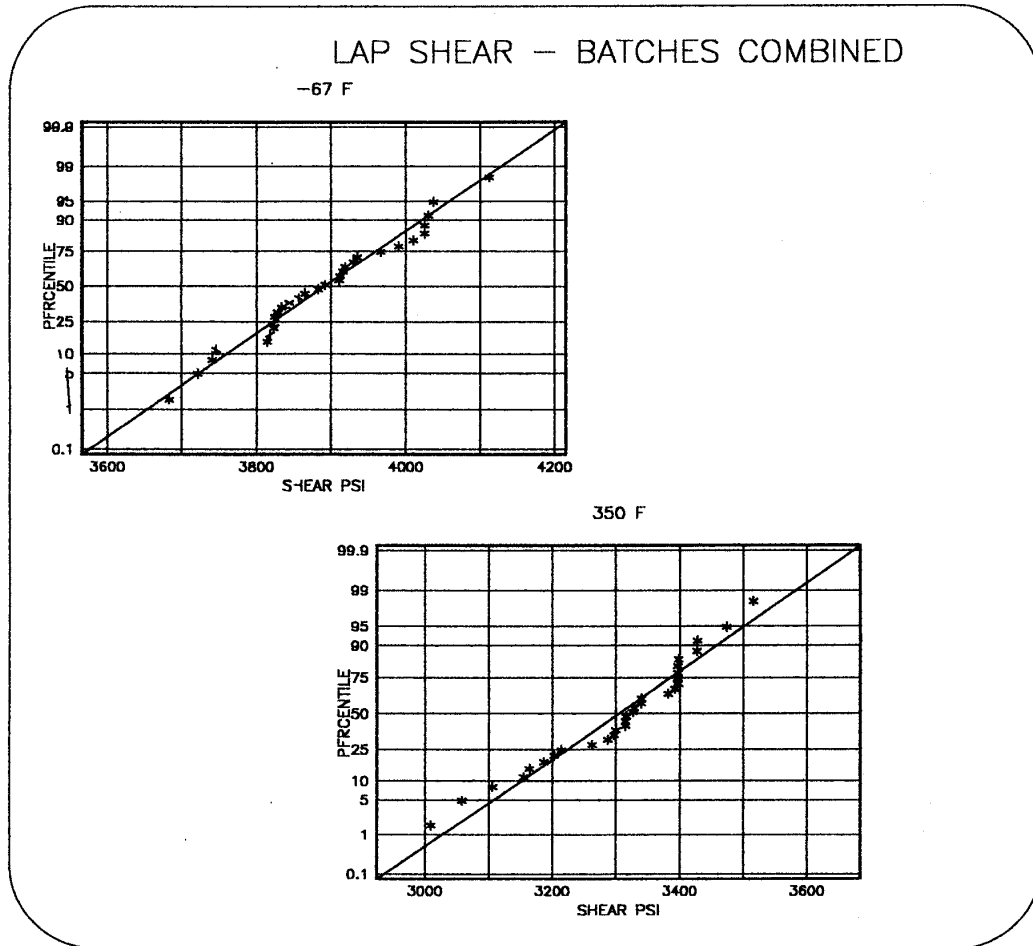
APPENDIX 3.1

LAP SHEAR / THICK ADHEREND (BATCHES COMBINED)**LEGEND**

LS = Lap Shear ; TA = Thick Adherend

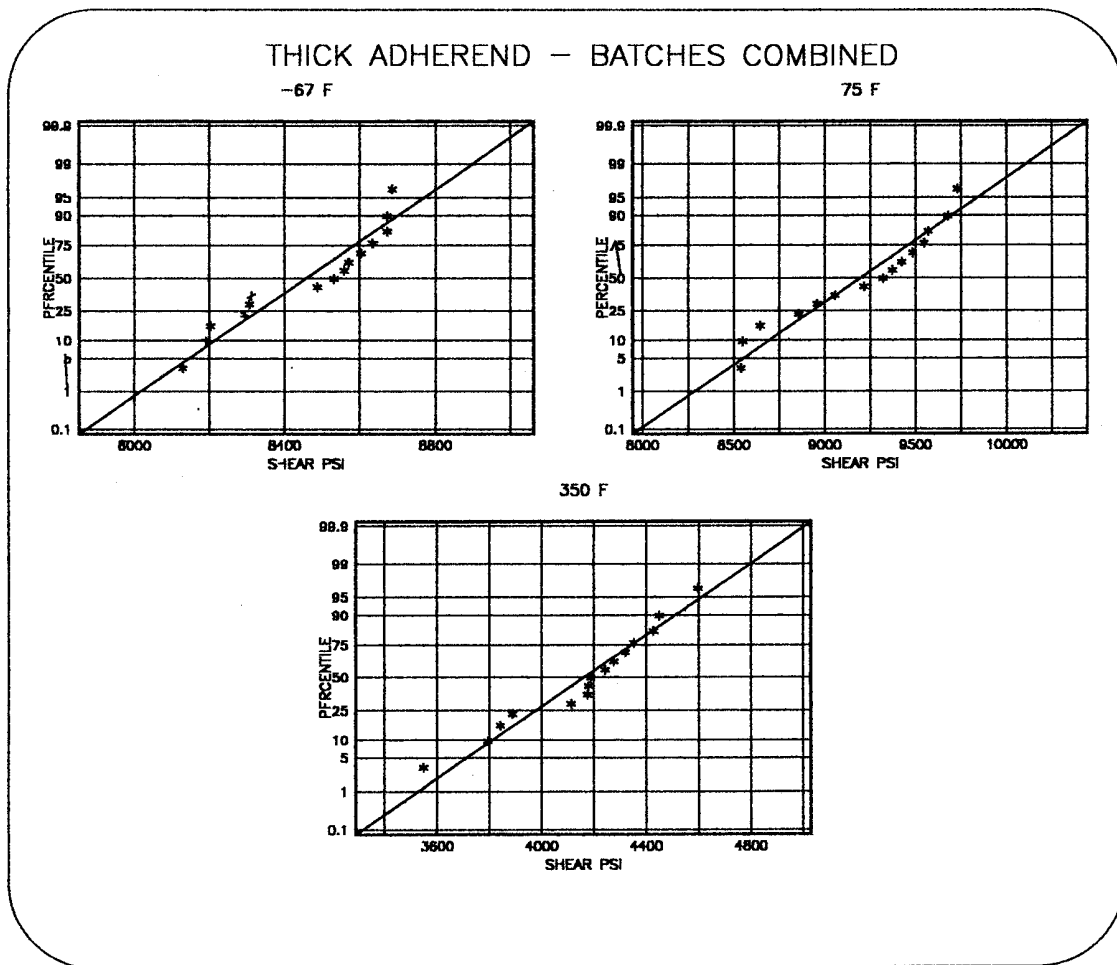
TEMP.	LAP SHEAR		THICK ADHEREND		LS v. TA COMPARISONS	
	AVERAGE	S.DEV.	AVERAGE	S.DEV	BARTLETT S.DEV. TEST	ANOVA AVERAGE TEST
-67 F	3890.5	103.8	8457.3	196.4	DIFF.	DIFF.
75 F	4299.7	142	9196.5	406.1	*	*
350 F	3304.3	121.5	4160	282.4	DIFF.	DIFF.

* 75 F included for general comparison only.(75F / LS batch effect)

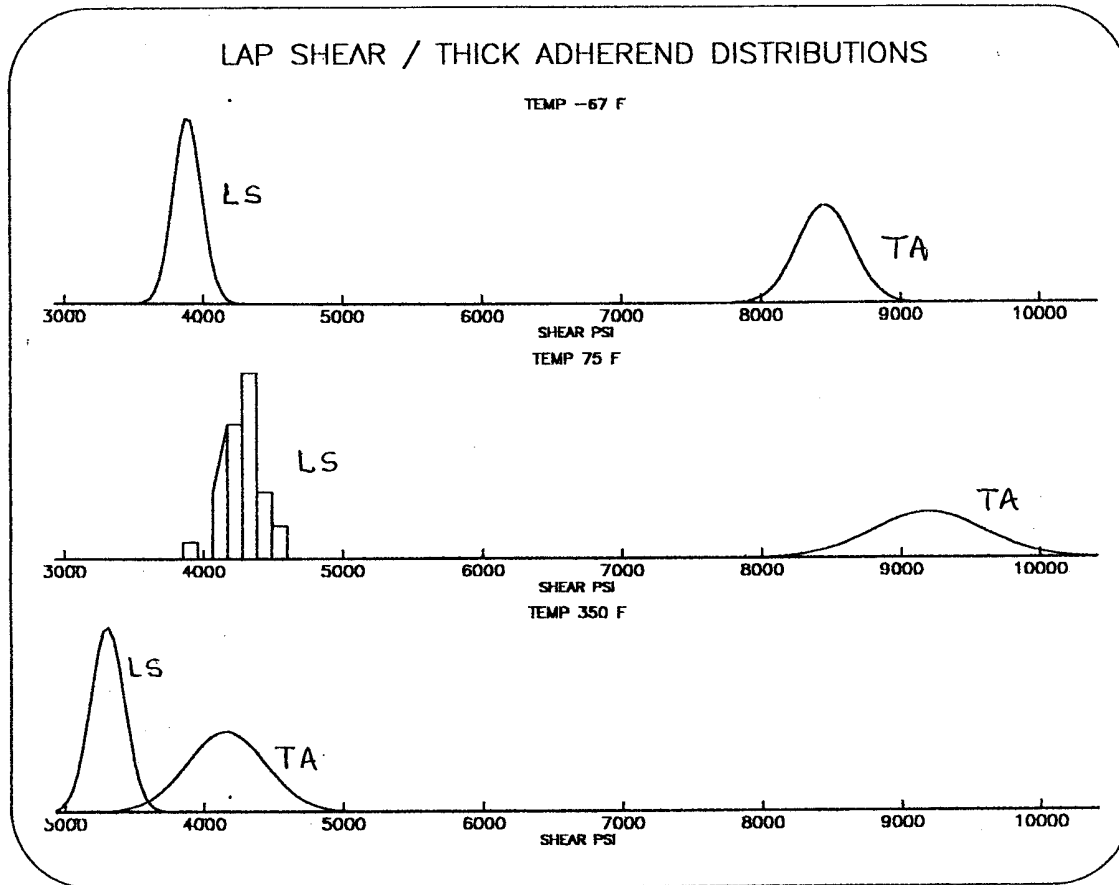
APPENDIX 3.2**LAP SHEAR DISTRIBUTION ANALYSIS**

TEMP.	AVERAGE	S.DEV	NORMAL DISTRIBUTION TEST (KOLMOGOROV-SMIRNOV)
- 67 F	3890.5	103.8	NORMAL
75 F	Test not performed due to batch differences		
350 F	3304.3	121.5	NORMAL

APPENDIX 3.3

THICK ADHEREND DISTRIBUTION ANALYSIS

TEMP.	AVERAGE	S.DEV	NORMAL DISTRIBUTION TEST (KOLMOGOROV-SMIRNOV)
- 67 F	8457.3	196.4	NORMAL
75 F	9196.5	406.1	NORMAL
350 F	4160	282.4	NORMAL

APPENDIX 4.1**LAP SHEAR / THICK ADHEREND DISTRIBUTIONS COMBINED****LEGEND.**

LS = LAP SHEAR

TA = THICK ADHEREND

TEMP.	LAP SHEAR		THICK ADHEREND		LS v TA COMPARISON
	AVERAGE	S.DEV.	AVERAGE	S.DEV	DIFF. IN AVERAGE
- 67 F	3890.5	103.8	8457.3	196.4	4566.8
75 F	4299.7	142	9196.5	406.1	4896.8
350 F	3304.3	121.5	4160	282.4	855.7

Case Study 3

Statistical Analysis of Thick Adherend Results

1. INTRODUCTION

Statistical analysis such as design of experiment (DOE) techniques can be used to establish the interactions between the critical factors that affect the durability of adhesive joints to hostile environments. A number of case studies have been used within the programme to demonstrate the use of these techniques in quantifying the relationship between the critical factors [1-2]. As part of the statistical validation element for PAJ3, an experimental programme was **designed, implemented and analysed** using DOE techniques.

The programme was set-up to evaluate the durability performance of 5251 aluminium alloy TAST tensile specimens immersed in distilled / deionised water at either 25°C, 40°C or 60°C for periods of up to 6 weeks. Batches of conditioned specimens (five specimens per batch) were to be withdrawn at selected intervals over a 6 week period (i.e. 0, 3, 7, 14, 21 and 42 days) and tested at room-temperature. The TAST specimens consisted of 5 mm thick pre-shaped adherends bonded with either AV119 (also known as Araldite® 2007), F241 or AF126-2. PTFE spacers were used to prevent the formation of an adhesive fillet at the ends of the overlap. Two surface pre-treatments were considered in the study.

A brief description of the adhesives and surface treatment used in the assessment are provided below.

Adhesives

Araldite 2007 (AV119): A one part epoxy adhesive supplied by Ciba Speciality Polymers. The cure schedule was 140 °C for 75 minutes.

F241: A toughened acrylic adhesive supplied by Permabond Adhesives Ltd. The adhesive was cured by applying an initiator to one surface and the acrylic resin to the other surface. The adhesive was allowed 24 hours to cure at room-temperature.

AF126-2: A modified epoxy film adhesive supplied by 3M, UK. The adhesive contains a carrier fabric for bondline thickness control. The cure cycle was 120 °C for 90 minutes.

Surface Treatments

Grit Blast + Degrease (GB): Prior to bonding, the adherends were degreased with 1,1,1-trichloroethane and then grit blasted using 80/120 alumina to produce an uniform matt

finish. A pressure of 85 psi was used to grit-blast the areas to be bonded. Any dust remaining after grit blasting was removed with clean compressed air. The surfaces to be bonded were then degreased again with 1,1,1-trichloroethane and dried. The time between bonding and surface pretreatment was approximately 1 hour.

Grit Blast + A187 (g-glycidoxypyltrimethoxy) Silane (GPS): The initial surface pretreatment was identical to the procedure employed for grit blast + degrease. The grit-blasted + degreased surface was subsequently coated with A187 silane coupling agent (supplied by Union Carbide). A 1% solution of A187 silane was prepared in deionised water and allowed to stand for 90 minutes to hydrolyse. The solution was brushed onto the prepared surfaces. The coated adherends were then positioned to drain off the excess solution and allowed to dry in air for 2 to 3 hours before applying the adhesive.

2. DESIGN OF EXPERIMENT

The study has been constructed in two stages in order to maximise the information which could be gained from the experiment and at the same time minimise the impact of spurious non-experimental or “noise” factors during the execution of the experiment.

Stage 1 Design the experiment to avoid bias and “noise” factors.

Stage 2 Analyse the data from the experiment and generate conclusions about the experimental factors.

Stage 1 of the process will be covered in this section with Stage 2 being handled in Section 3.

2.1 EXPERIMENTAL FACTORS

(A) Adhesive (x 3): AV119, F241 and AF126-2

(S) Surface treatment (x 2): Grit blast and A187 silane

(C) Temperature (x 3): 25°C, 40°C and 60°C

(T) Exposure Time (x 6): 0, 3, 7, 14, 21 and 42 days

Each condition is to be replicated five (5) times.

2.2 INITIAL DATA REVIEW

A full factorial design is required in order to investigate all combinations of the factors mentioned above (see Table 1).

Table 1 Experimental Design Matrix

Temperature (°C)	Time (days)	Surface Treatment					
		Grit Blast			A187 Silane		
		Adhesive			Adhesive		
		AV119	F241	AF126-2	AV119	F241	AF126-2
25	0						
	3						
	7						
	14						
	21						
	42						
40	0						
	3						
	7						
	14						
	21						
	42						
60	0						
	3						
	7						
	14						
	21						
	42						

The matrix structure in Table 1 has been arranged so that columns correspond to specimen conditions and rows correspond with environmental conditions. The number of specimens required for the experimental programme is determined by:

(3 adhesives) x (2 surface treatments) x (3 temperatures) x (6 exposure times) x (5 replicates)

= **540 specimens**

2.3 EXPERIMENTAL PROCESS AND CONSTRAINTS

The experimental matrix in Table 1 showing the data required does not show how the data is to be collected (i.e. the order in which the various experimental conditions are to be performed). It is important that the experimental sequence employed nullifies the effect of “noise” factors, those which are not part of the group included as formal factors as defined in Section 2.1, on the results and the conclusions.

Appendix 1 shows the sequence of activity involved in producing the specimens, the environmental conditioning and the test approach, thus showing the process by which the experimental conditions will be achieved. It also provides certain key information, adjacent to each process element, relating to the number of specimens that can be

processed at one time and in the case of the environmental conditioning stage, the number of specimens that can be processed at each temperature. This information will be used in designing the experimental approach.

2.4 EXPERIMENTAL APPROACH / PLAN

2.4.1 Experimental Approach

There are three approaches used in designing experiments to nullify the impact of “noise” factors:

1. Blocking (specifically identifying the “noise” factor involved, and arranging small balanced sets of experiments for each “noise” factor level).
2. Accounting for the “noise” factor by labelling the samples with the “noise” factor level. Subsequent analysis used to untangle the effect of the “noise” factor.
3. Randomising (experiment conditions are performed in a random order).

Approaches 2 and 3 will be used for selecting specimens and environmental conditioning.

Cut Panels: The 540 specimens, required for the complete experiment, are to be cut from two panels of aluminium which are approximately 1 m square. The specimens consist of two pre-shaped sections that are 110 mm long and 25 mm wide. To avoid the possibility of a panel effect (i.e. differences in material properties between the two panels), each batch of specimens will contain specimens from panels labelled “A” and “B”. Since each experimental condition requires five (5) replicates, each replicate will be made up of 3 specimens from panel “A” and 2 specimens from panel “B”.

Environmental Conditioning: The main constraint on the experiment is the number of specimens that can be accommodated at this stage (i.e. the number of specimens that can be environmentally conditioned at any one time). An experimental sequence therefore needs to account for this constraining factor when determining which specimen surface treatment/adhesive type combinations are to be present in the environmental conditioning stage at any time. The sequence of environmentally conditioning the specimens will be randomised.

It can be seen from the process chart in Appendix 1 that there are three baths with temperatures set at 25°C, 40°C and 60°C, each containing three beakers into which 15 specimens can be placed. Since five replicates at each condition are to be tested, three experimental combinations can be placed in each beaker. The schematic diagram in Appendix 2 shows such a sequence where the exposure times and surface treatment/adhesive combinations have been randomly selected.

The full experimental sequence, shown in Appendix 3, is appropriate for each of the conditioning temperatures 25°C, 40°C and 60°C. It can be seen that the first entries are those shown in Appendix 2. On completion of the environmental conditioning stage, each batch of specimens are to be replaced by the next combination on the list. It would be entirely possible to exercise a greater level of randomisation in the experiment structure, but a pragmatic cut-off was used to give a good balance between controlling “noise” and managing the experiment (**NB. Process sequence needs to be practical**).

2.4.2 Specimen Preparation

The sequence shown in Appendix 3 is that required at the environmental stage of the process. A project plan needs to be constructed which translates this sequence into a time schedule for specimen preparation (i.e. surface treatment, adhesive preparation, joint manufacture and cure stages). Care should be exercised to ensure adherence at all times to the process conditions at each of the stages and that inter-stage timings are maintained throughout the programme. This phase is also geared towards avoiding the possible inclusion of other “noise” factors in the experiment.

At the Joint Manufacturing stage, there is provision for six specimens to be bonded in each bonding fixture. Since only five specimens are needed for each experimental combination then five positions need to be identified for use throughout the programme. There is a possibility that the clamping forces vary across the fixture.

2.4.3 Time Data Recording

It is recommended that a start date be identified and that all events subsequent to the date be labelled as Day “X” where “X” is the number of days from the start date. This will enable the collection of supplementary data as indicated below.

2.5 DATA COLLECTION PLAN

Although Appendix 1 contains the essence of the experimental results in terms of the formal factors included in the experiment, an alternative layout is needed for data collection in order that other data can be included and used in the analysis phase. Appendix 4 contains the template for data collection. The template includes provision for panel “A” and “B” information and for information on inter-process stages. The list is not exhaustive, and if other information is required to be collected by the programme manager, further columns can be added as needed. It is suggested that some caution is exercised in including too many new factors so as to avoid information overload and analysis complexity. It is recommended that the layout shown in Appendix 4 be inserted into a spreadsheet for the analysis stage of the programme.

3. EXPERIMENTAL STRUCTURE

This section analyses the results obtained from the experimental programme formulated in Section 2. The factors and levels investigated in the experimental programme are shown in Section 2.1. It was necessary to use three panels (**P**), labelled A, B and C, to produce the 540 specimens. Joint strength data measured using the TAST is given in kN.

3.1 INITIAL DATA ANALYSIS

The experimental results are summarised in Appendix 5. Appendix 5 shows the average and standard deviation for each test condition based on five replicates.

3.1.1 Thick Adherend Comparisons

The six charts in Appendix 6 show the average values of all the test conditions. Each chart contains the results for the three adhesives assessed in the programme over the exposure time range (0 to 42 days) for each of the temperature and surface treatment combinations. Each graph was constructed with a common vertical scale to make visual assessment easier. It is clear that there are differences between the results over the different conditions and the relationships do not hold true over the full exposure time range. More detailed analysis is required to untangle the relationships.

3.1.2 Relationship Between Mean And Standard Deviation

A more thorough analysis requires that the standard deviations across the test conditions be consistent or homogeneous and unrelated to the test average (or mean) at that condition. The top chart in Appendix 7 shows a plot of 108 test means against their standard deviations. It appears from this plot that as the mean increases, the standard deviation decreases, thus contravening one of the conditions required. This relationship is investigated further in the lower plot of Appendix 7 where log mean is plotted against the log standard deviation. Superimposed on this plot is the “best fit” regression line which has a negative slope. The formal analysis of the relationship is shown in the Regression Coefficients Analysis table in Appendix 7. The calculated value of the slope is -1.272 with 95% confidence limits covering the range -1.916 to -0.628. The *t*-value for the slope indicates that there is a significant slope to the data.

In summary, this all points to a significant effect with the standard deviations being related to the mean. The significance of this is that further analysis of the original data would be inadvisable. Some transformation of the response data will be required before proceeding further with the analysis. The relationship shown by the slope of -1.272 enables a suitable transformation to be developed. Since the confidence interval covers the value -1, this will be used to produce the simplest structure for the transformation. The theory for this relates the slope of the line to a power type of transformation. A slope of -1 equates to a power of 2, or the square of the original data. Hence, all further analysis will be constructed using TA^2 where TA = Thick Adherend.

3.2 ANALYSIS AND MODEL BUILDING

The formal analysis was constructed using Analysis of Variance (ANOVA). The significance of the effects shown by the ANOVA was further investigated using Analysis of Means (ANOM). The “goodness” of fitted model from the above analyses was further developed through investigating the residuals from the fitted model and also by comparing the actual results against those predicted through the use of the model.

3.2.1 ANOVA and Model Residual Analysis

The ANOVA chart (Appendix 8) shows only the significant effects. It can be seen that temperature (**C**), Time (**T**) and Surface Treatment (**S**) are the significant main effects. In addition, there are several first order interactions (**CT**, **CS**, **CA**, **CP**, **TA** and **SA**) and second order interactions (**CTS**, **CTA**, **TSA** and **CSA**). The net effect is that the structure linking the TAST results to the experimental factors is very complex. The model fitted explains 69% of the observed variation in the results. The absence of adhesives (**A**), from the significant main effects does not mean that this is not an important factor. It just implies that the effect of adhesives is more marked in combination with other factors as can be seen from the significant first and second order interactions identified above.

The Residual Analysis chart in Appendix 8 is a very useful tool. It assists in verifying whether the transformation used (TA^2) is reasonable. If this is the case then the distribution of Residuals where Residual = Actual - Predicted should follow an approximately Normal distribution. It can be seen from the density function and probability plot that the distribution of the Residuals closely follows a Normal distribution. Hence, the use of the TA^2 transformation is reasonable. This means that a model based on this transformation can be safely analysed through ANOVA and ANOM methods.

3.2.2 Analysis of Means (ANOM)

The charts in Appendices 9, 10 and 11 show the ANOM analysis for each of the significant effects shown by the ANOVA. Appendix 9 (ANOM plots) shows the main effects of **C**, **T**, **S**, **A** and **P**. It can be seen that Time, Temperature and Surface Treatment show significant effects with adhesive and Panel showing much less of an effect. There is progressive deterioration in strength as temperature is increased from 25 °C to 60 °C with the most marked change being between 25 °C and 40 °C. There is also a progressive deterioration in strength as exposure time is increased with the most marked change occurring between 0 and 3 days. The surface treatment has a large effect on strength with A187 silane showing a significantly larger strength compared to the grit blast treatment.

Appendix 9 also shows the interaction analysis of first order interactions **CT**, **CS** and **CA** as three separate plots within the chart. The **CT** effect is shown through the change in strength over the exposure time of 0 to 42 days with the effect being different across each of the temperatures of 25 °C, 40 °C and 60 °C. The **CS** effect shows the difference in strength in changing from grit blast to A187 silane surface treatment is different for each temperature. The **CA** effect shows that the difference in strength in changing adhesives is not the same across each temperature; in fact there is a marked effect at 25 C where F241

gives a much lower strength than either AV119 or AF126-2. This effect is not found at 40 °C and 60 °C.

Appendix 10 shows the interaction analysis for the first order interactions **CP**, **TA** and **SA**. The **CP** effect shows through the change in strength between panels with the effect being different across the temperatures.. This effect is very marked when comparing the results for 25 °C with the strengths measured at the other two temperatures. The **TA** effect shows that the change across the adhesives is not the same at each exposure time. This is most marked at 0 and 42 days where it can be seen that the F241 adhesive gives a much lower strength relative to the other adhesives. The **SA** effect shows that the change in strength across the adhesives is not the same for each surface treatment.

Appendix 10 also shows the interaction analysis for **CTA**, which represents an interaction between **C**, **T** and **A**. Interactions at this level are usually more difficult to interpret. The essence of this interaction is that the change in strength across the adhesives for each of the exposure times 0 to 42 days is not the same for each temperature. Again, adhesive F241 behaves differently at 0 and 42 days compared with the other exposure times, but this interaction also shows a temperature contribution.

Appendix 11 shows the interaction between **CTS** and **CSA**. The **CTS** effect shows through the change in strength between the two surface treatments for each exposure time is not the same for each temperature. This is most marked at 60 °C and exposure times of 7 and 14 days where it can be seen that the change from grit blast to A187 silane is different to other combinations. The **CSA** effect shows through the change in strength across the adhesives for each surface treatment is different for each temperature. Appendix 11 also shows the interaction analysis for **TSA**. The analysis shows that the change in strength across the adhesives for each surface treatment is not the same for each time period.

3.3 Model and Residual Analysis

As part of the model verification it is worthwhile investigating the behaviour of the residuals. Appendix 12 shows the plot of the residuals against each of the 540 observations. There is no pattern of residual values which seem to be related to the observation number and thereby being associated with particular combinations of experimental factors. It can therefore be concluded that the model is reasonable over the whole experimental space.

Appendix 12 also shows a plot of the observed TAST load values compared with predicted values from the model. These values have been converted back to the untransformed scale. It can be seen that there is a strong band of results which demonstrate the equivalence of the model fitted values and the measured values. Most of the variation shown by the band is represented by the variation found during the five repeat measurements taken at each test point. There are a few points at the lower end of the plot which show a divergence from the overall pattern. These individual results could benefit from further examination, but these results do not negate the overall success of the model in predicting the TAST test data.

An alternative view of the measured versus predicted results is shown in Appendix 13. Appendix 13 shows two plots of measured values versus observation number and model fitted values versus observation number. It can be seen from the two plots that the overall pattern of the predicted values follows the measured values very closely. The only divergence's are in some specific cases where some very low values were recorded.

4. CONCLUSIONS

The following conclusions can be drawn from the analyses:

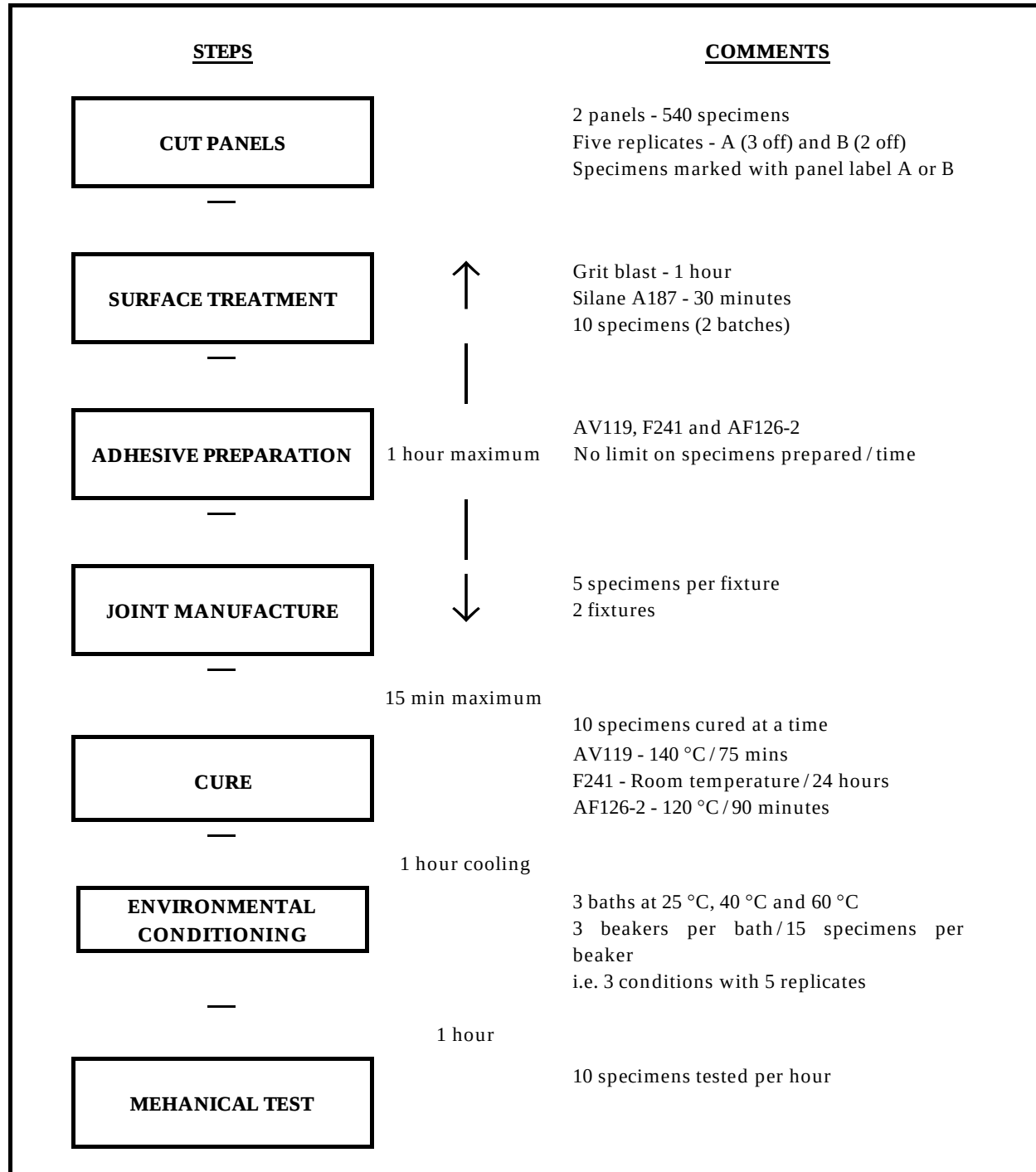
1. The statistical tools used have proved very useful in analysing a complex process.
2. The factors of Temperature, Exposure Time and Surface Treatment have a significant impact on the thick-adherend strength of the joint:
 - i) Joint strength decreases with increasing conditioning temperature.
 - ii) Joint strength decreases with increasing exposure time.
 - iii) A187 silane treatment resulted in significantly higher joint strengths compared with grit blasting.
 - iv) The adhesive type was important, but only in combination with other factors as an interaction.
3. The system investigated contained a great deal of interaction between the factors. First and second level interactions were found to have a significant effect. This has meant that the predictive model developed to represent the system is very complex. The model, however, explained over 69% of the overall variation in the results; a very reasonable performance.
4. In some cases, a transformation of the original data will be needed to enable meaningful analysis to proceed. In this case a power transformation of TA^2 was needed.
5. There are many ways that the experiment could have been run to investigate the combinations of factors. The use of "Design of Experiments" thinking, incorporating an understanding of the process of producing test specimens has provided a powerful platform from which the conclusions can be viewed with confidence.

REFERENCES

1. Olusanya, A., Tully, K, Mera, R. and Broughton, W.R., "A Comparison of Commercial Design of Experiment Software Programs for the Analysis of Durability Data", NPL Report CMMT(A) 98, 1998.
2. Olusanya, A., "The Use of Design of Experiments Techniques to Determine the Relative Effectiveness of Silane Coupling Agents on the Durability of Titanium Alloy Joints. A Case Study", NPL Report CMMT(A) 128, 1998.

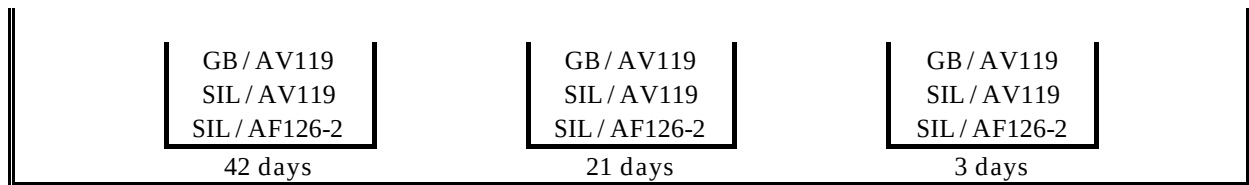
ADDITIONAL READING

1. Box, Hunter and Hunter, "Statistics for Experimenters", Wiley.
2. Montgomery, Design and Analysis of Experiments", Wiley.
3. Grove and Davis, "Engineering Quality and Experimental Design", Longman.
4. Draper and Smith, "Applied Regression Analysis, Wiley.
5. Kleinbaum, Kupper and Muller, "Applied Regression Analysis and Other Multivariable Methods", PWS-Kent.

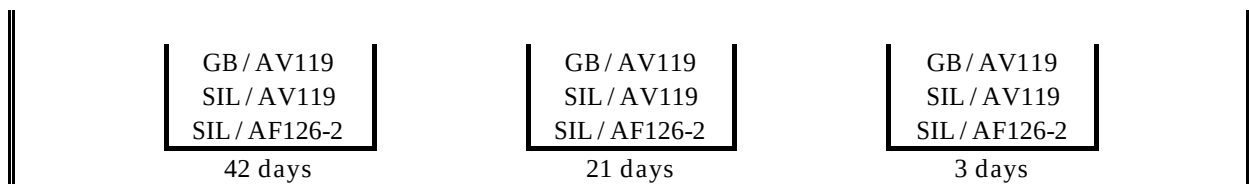
APPENDIX 1**PROCESS SEQUENCE**

APPENDIX 2

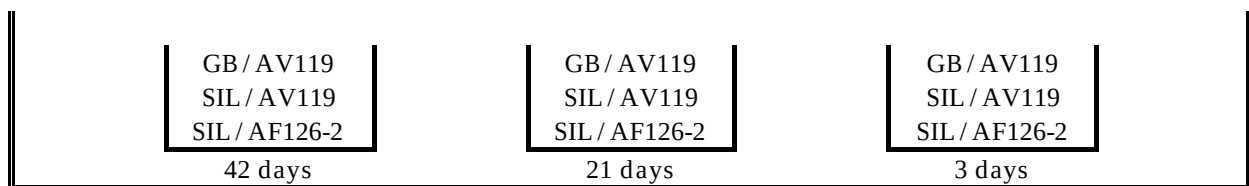
ENVIRONMENTAL CONDITIONING EXAMPLE (BATHS/BEAKERS/TEST COMBINATION LAYOUT)



25 °C



40 °C



60 °C

Notes

GB = GRIT BLAST; SIL = A187 SILANE

APPENDIX 3

**EXPERIMENT SEQUENCE
(AT ENVIRONMENTAL CONDITIONING STAGE)**

Time (days)	Surface Treatment	Adhesive Type
42	GB	AV119
	SIL	AV119
	SIL	AF126-2
21	GB	AV119
	SIL	AV119
	SIL	AF126-2
3	GB	AV119
	SIL	AV119
	SIL	AF126-2
14	GB	AV119
	SIL	AV119
	SIL	AF126-2
7	GB	AV119
	SIL	AV119
	SIL	AF126-2
0	GB	AV119
	SIL	AV119
	SIL	AF126-2
42	GB	F241
	GB	AF126-2
	SIL	F241
21	GB	F241
	GB	AF126-2
	SIL	F241
3	GB	F241
	GB	AF126-2
	SIL	F241
14	GB	F241
	GB	AF126-2
	SIL	F241
7	GB	F241
	GB	AF126-2
	SIL	F241
0	GB	F241
	GB	AF126-2
	SIL	F241

Notes

Each combination to be produced for each temperature 25 °C, 40 °C and 60 °C.
Each combination to have five replicates produced.

APPENDIX 4

DATA COLLECTION PLAN

[illegible]

Notes

1. As an example, the 15 specimens from the first three experiment combinations shown in Appendix 3 are included.
2. Day No. ST/Adh is the day number when the specimens were prepared with surface treatment (ST)/ Adhesive (Adh) combination.
3. Day No. Env.Con is the day number when the specimens were placed into environmental conditioning.
4. Day No. Mech.Test is the day number when the specimens were tested for Mechanical strength.
5. A comments column is included to allow the experimenter to note other facts or observations which are experienced as the experiment progresses.

APPENDIX 5

DIX 5

THICK ADHEREND TEST DATA

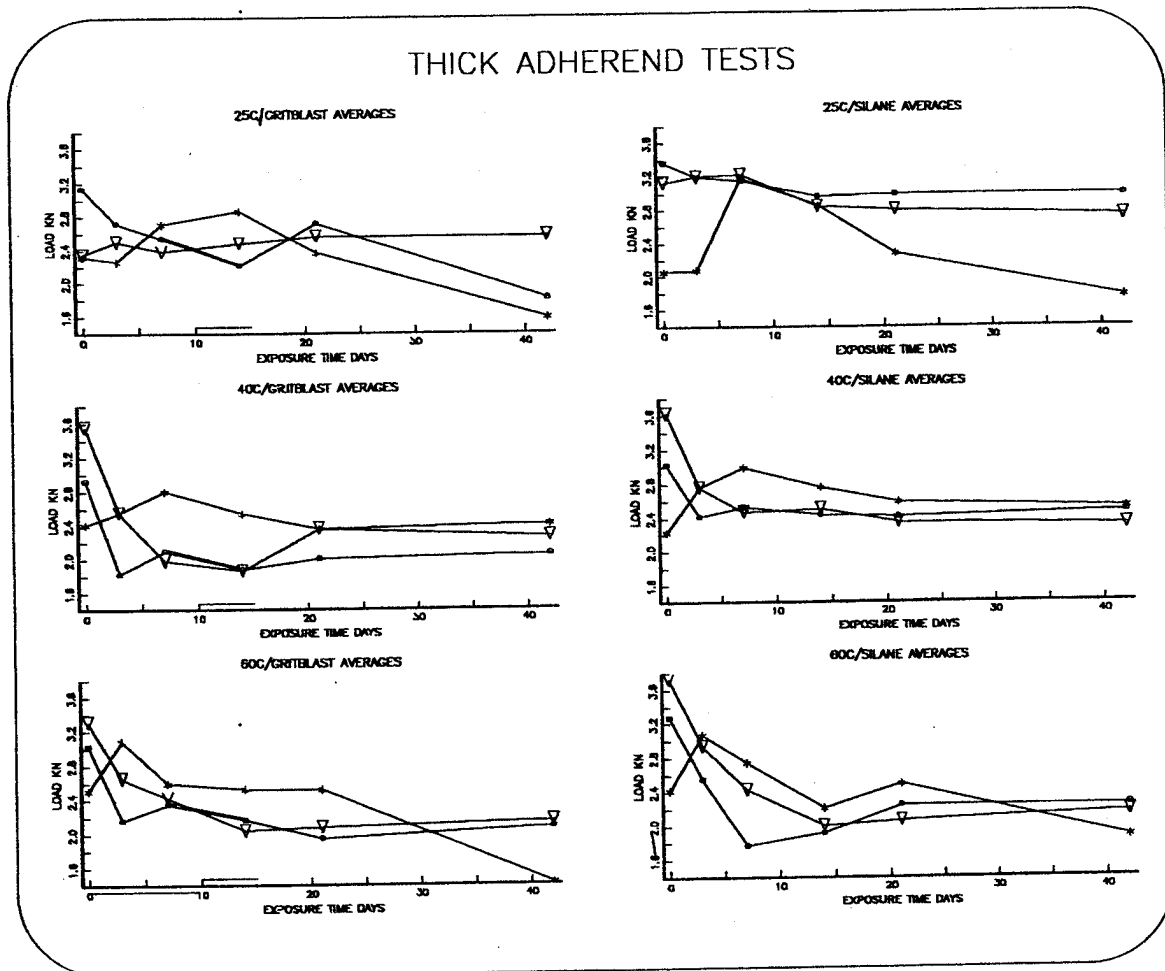
(Data shown are based on 5 results at each test point)

TEMP 25 deg C					TEMP 40 deg C					TEMP 60 deg C				
EXPOSURE TIME (DAYS)	SURFACE TREATMENT	ADHESIVE	LOAD KN AVERAGE	STD DEV	EXPOSURE TIME (DAYS)	SURFACE TREATMENT	ADHESIVE	LOAD KN AVERAGE	STD DEV	EXPOSURE TIME (DAYS)	SURFACE TREATMENT	ADHESIVE	LOAD KN AVERAGE	STD DEV
0	GRIT BLAST	AV119	3.136	0.075	0	GRIT BLAST	AV119	2.925	0.120	0	GRIT BLAST	AV119	3.028	0.195
0	GRIT BLAST	F241	2.319	0.178	0	GRIT BLAST	F241	2.415	0.290	0	GRIT BLAST	F241	2.514	0.189
0	GRIT BLAST	AF126	2.346	1.145	0	GRIT BLAST	AF126	3.551	0.211	0	GRIT BLAST	AF126	3.311	0.151
0	SILANE	AV119	3.372	0.113	0	SILANE	AV119	3.028	0.199	0	SILANE	AV119	3.275	0.208
0	SILANE	F241	2.059	0.646	0	SILANE	F241	2.238	0.150	0	SILANE	F241	2.424	0.180
0	SILANE	AF126	3.135	0.195	0	SILANE	AF126	3.630	0.212	0	SILANE	AF126	2.727	0.239
3	GRIT BLAST	AV119	2.724	0.370	3	GRIT BLAST	AV119	1.829	0.877	3	GRIT BLAST	AV119	2.166	0.167
3	GRIT BLAST	F241	2.261	0.597	3	GRIT BLAST	F241	2.552	0.460	3	GRIT BLAST	F241	3.092	0.153
3	GRIT BLAST	AF126	2.501	0.156	3	GRIT BLAST	AF126	2.552	0.460	3	GRIT BLAST	AF126	2.651	0.534
3	SILANE	AV119	3.198	0.039	3	SILANE	AV119	2.423	0.758	3	SILANE	AV119	2.554	0.398
3	SILANE	F241	2.080	0.946	3	SILANE	F241	2.763	0.251	3	SILANE	F241	3.076	0.153
3	SILANE	AF126	3.205	0.177	3	SILANE	AF126	2.763	0.251	3	SILANE	AF126	2.937	0.417
7	GRIT BLAST	AV119	2.544	0.158	7	GRIT BLAST	AV119	2.100	0.261	7	GRIT BLAST	AV119	2.353	0.526
7	GRIT BLAST	F241	2.713	0.202	7	GRIT BLAST	F241	2.807	0.245	7	GRIT BLAST	F241	2.598	0.484
7	GRIT BLAST	AF126	2.382	0.593	7	GRIT BLAST	AF126	1.982	0.399	7	GRIT BLAST	AF126	2.425	0.348
7	SILANE	AV119	3.154	0.071	7	SILANE	AV119	2.532	0.137	7	SILANE	AV119	1.773	0.421
7	SILANE	F241	3.187	0.095	7	SILANE	F241	2.994	0.148	7	SILANE	F241	2.745	0.286
7	SILANE	AF126	3.230	0.428	7	SILANE	AF126	2.474	0.769	7	SILANE	AF126	2.429	0.374
14	GRIT BLAST	AV119	2.214	0.562	14	GRIT BLAST	AV119	1.881	0.305	14	GRIT BLAST	AV119	2.168	0.135
14	GRIT BLAST	F241	2.867	0.204	14	GRIT BLAST	F241	2.544	0.105	14	GRIT BLAST	F241	2.530	0.321
14	GRIT BLAST	AF126	2.482	0.524	14	GRIT BLAST	AF126	1.865	0.250	14	GRIT BLAST	AF126	2.040	0.333
14	SILANE	AV119	2.969	0.297	14	SILANE	AV119	2.441	0.304	14	SILANE	AV119	1.921	0.922
14	SILANE	F241	2.861	0.183	14	SILANE	F241	2.766	0.227	14	SILANE	F241	2.215	1.075
14	SILANE	AF126	2.846	0.476	14	SILANE	AF126	2.512	0.410	14	SILANE	AF126	2.003	0.277
21	GRIT BLAST	AV119	2.723	0.191	21	GRIT BLAST	AV119	2.009	0.061	21	GRIT BLAST	AV119	1.946	0.165
21	GRIT BLAST	F241	2.366	0.351	21	GRIT BLAST	F241	2.362	0.301	21	GRIT BLAST	F241	2.521	0.223
21	GRIT BLAST	AF126	2.564	0.447	21	GRIT BLAST	AF126	2.362	0.412	21	GRIT BLAST	AF126	2.079	0.235
21	SILANE	AV119	2.990	0.191	21	SILANE	AV119	2.419	0.261	21	SILANE	AV119	2.249	0.310
21	SILANE	F241	2.280	0.435	21	SILANE	F241	2.590	0.091	21	SILANE	F241	2.496	0.272
21	SILANE	AF126	2.803	0.592	21	SILANE	AF126	2.353	0.397	21	SILANE	AF126	2.065	0.254
42	GRIT BLAST	AV119	1.814	0.886	42	GRIT BLAST	AV119	2.053	0.079	42	GRIT BLAST	AV119	2.077	0.171
42	GRIT BLAST	F241	1.582	0.615	42	GRIT BLAST	F241	2.409	0.145	42	GRIT BLAST	F241	1.420	0.391
42	GRIT BLAST	AF126	2.570	0.436	42	GRIT BLAST	AF126	2.262	0.221	42	GRIT BLAST	AF126	2.145	0.149
42	SILANE	AV119	2.986	0.218	42	SILANE	AV119	2.478	0.240	42	SILANE	AV119	2.251	0.128
42	SILANE	F241	1.763	0.691	42	SILANE	F241	2.522	0.237	42	SILANE	F241	1.875	0.195
42	SILANE	AF126	2.734	0.490	42	SILANE	AF126	2.319	0.151	42	SILANE	AF126	2.170	0.194

APPENDIX 6

THICK ADHEREND COMPARISONS

(TEMP / TIME / SURFACE / ADHESIVE COMBINATIONS)
(AVERAGE LOAD IN kN)

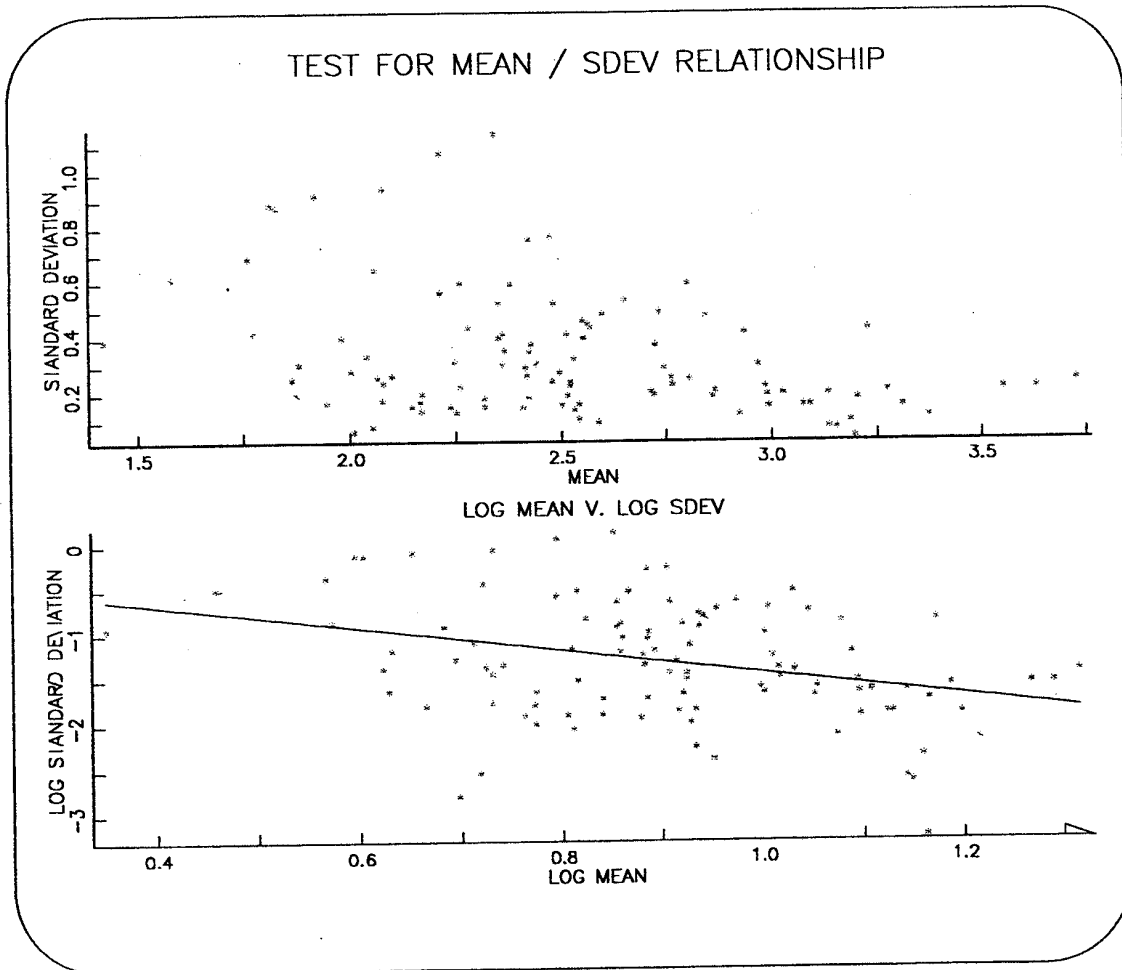


NOTES.

EACH OF THE 3 LINES ON EACH CHART REPRESENTS A DIFFERENT ADHESIVE WITH THE FOLLOWING SYMBOL CODING :

(◯ = AV119 ; * = F241 ; ▽ = AF126)

APPENDIX 7

TEST FOR RELATIONSHIP MEAN TO STANDARD DEVIATION**REGRESSION COEFFICIENTS ANALYSIS**

COEFFICIENT	COMPUTED VALUE	t VALUE FOR SIGNIFICANCE	95% CONFIDENCE INTERVAL FOR COEFFICIENT VALUE	
			LOWER	HIGHER
INTERCEPT	0.160	-0.547	-0.760	0.431
SLOPE	-1.272	-3.914	-1.916	-0.628

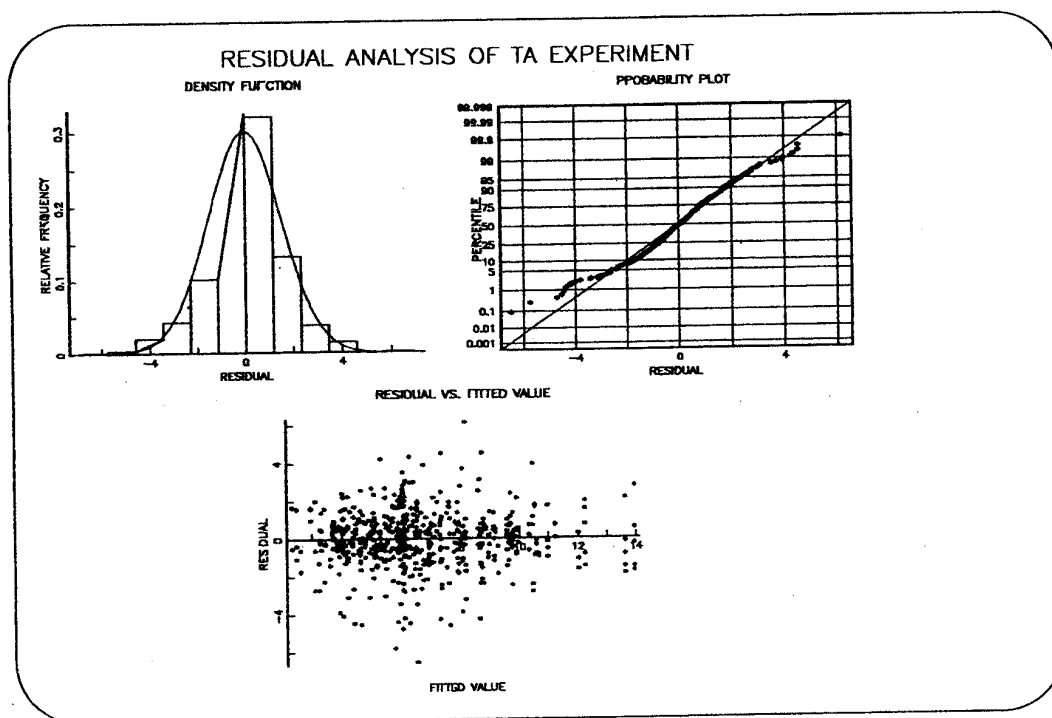
NOTES.

1. The straight line on the log mean v. log standard deviation plot, is the "best fit" regression line of the form $Y = a + bX$
2. The "t-value for significance" establishes whether the computed value of the coefficients for intercept and slope are significant

APPENDIX 8

ANOVA AND MODEL RESIDUAL ANALYSIS
(SIGNIFICANT EFFECTS ONLY)

ANALYSIS OF VARIANCE					
SOURCE	DF	SS	MS	F RATIO	SIG. LEVEL
C TEMPERATURE	2	124.19	62.09	22.42	5.2 E-10
T TIME	5	823.49	164.7	59.47	0
S SURFACE	1	204.26	204.26	73.75	6 E-13
CT	10	291.13	29.11	10.51	1.6 E-15
CS	2	55.18	27.59	9.96	5.8 E-5
CA	4	264.23	66.06	23.85	2.2 E-16
CP	3	73.93	24.64	8.89	1.7 E-5
TA	10	720.97	72.10	26.03	0
SA	2	51.00	25.50	9.21	1.2 E-4
CTS	10	64.07	6.41	2.31	1.1 E-2
CTA	20	309.38	15.47	5.58	5.9 E-13
TSA	10	53.33	5.33	1.93	4 E-2
CSA	4	27.48	6.87	2.48	4.3 E-2
ERROR	456	1263	2.77		
LACK OF FIT	84	181.42	2.16	0.74	0.95
PURE ERROR	372	1081.6	2.91		
TOTAL	539	4086			

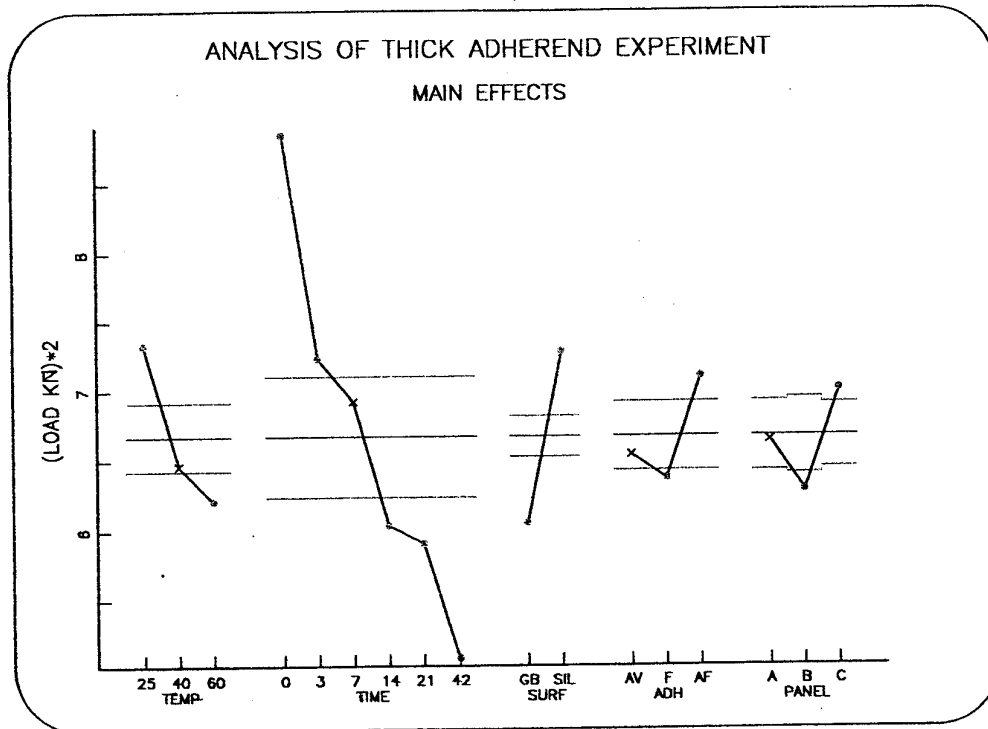
**NOTES**

C = TEMP ; T = TIME ; S = SURFACE ; A = ADHESIVE ; P = PANEL

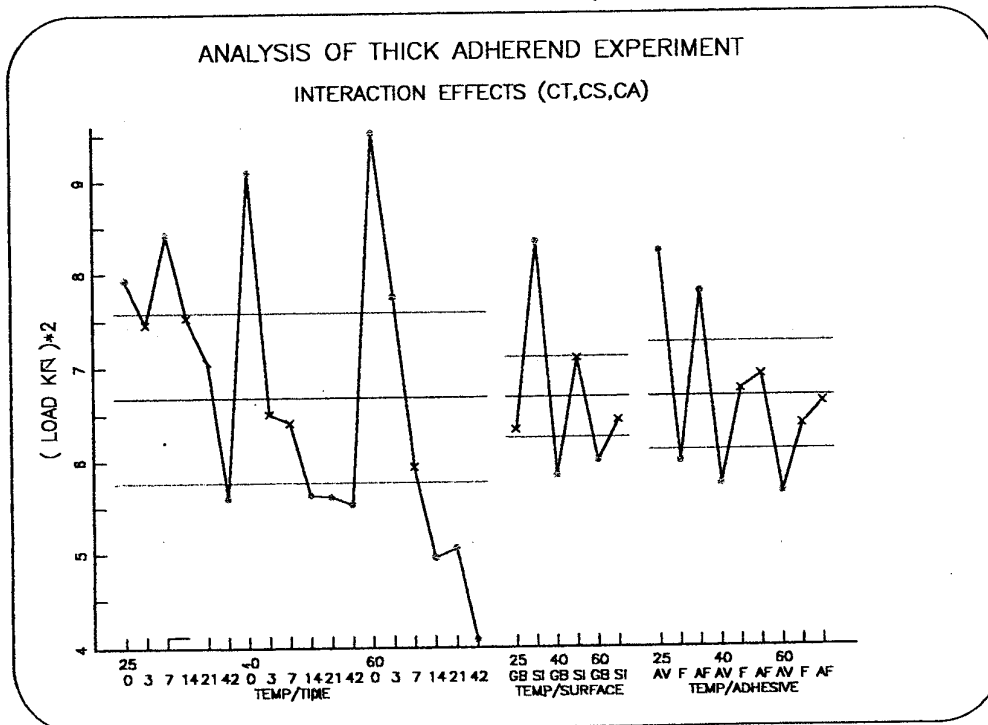
APPENDIX 9

ANALYSIS OF MEANS

MAIN EFFECTS (C, T, S, A, P)



INTERACTION EFFECTS (CT, CS, CA)

NOTES.

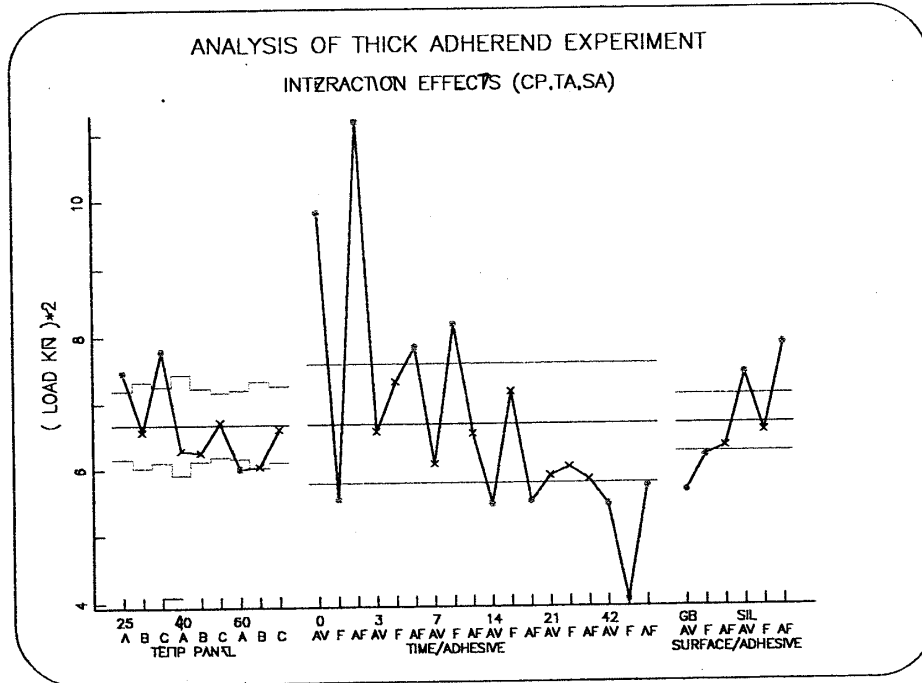
GB = GRIT BLAST ; SIL = SILANE

AV = AV119 ; F = F241 ; AF = AF126

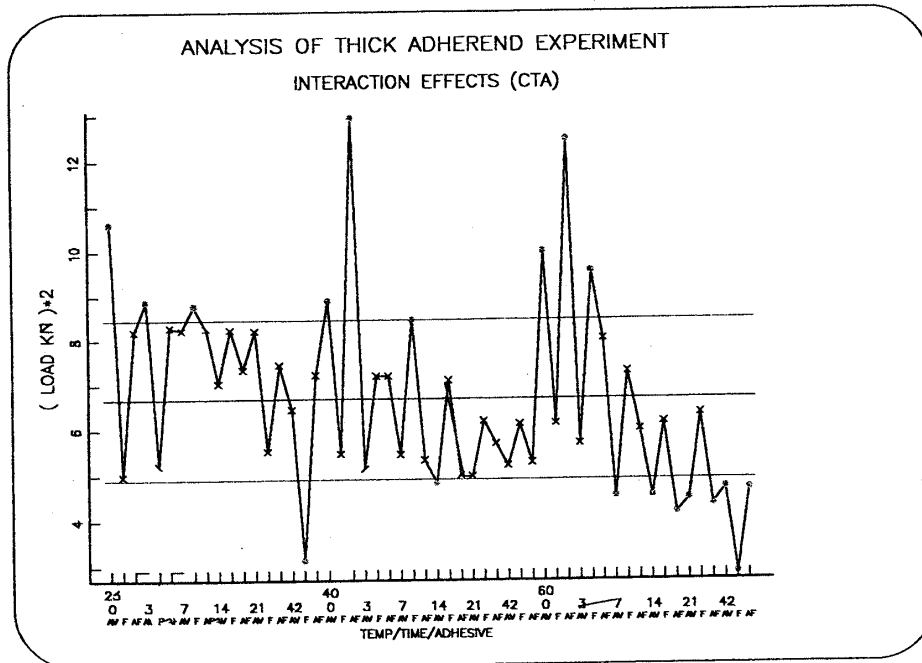
APPENDIX 10
APPENDIX 10

ANALYSIS OF MEANS

INTERACTION EFFECTS (CP, TA, SA)



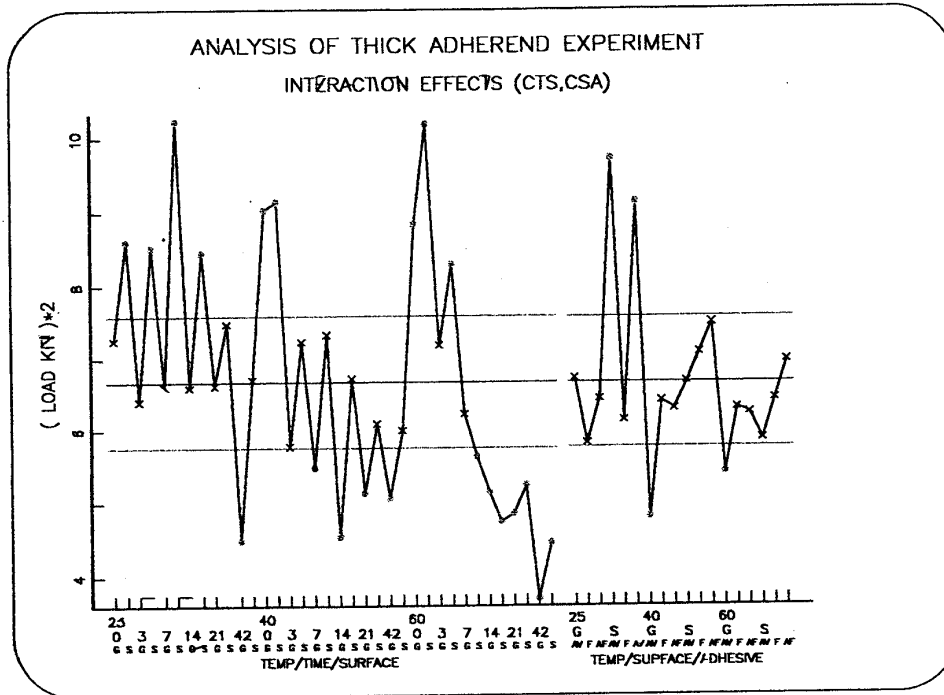
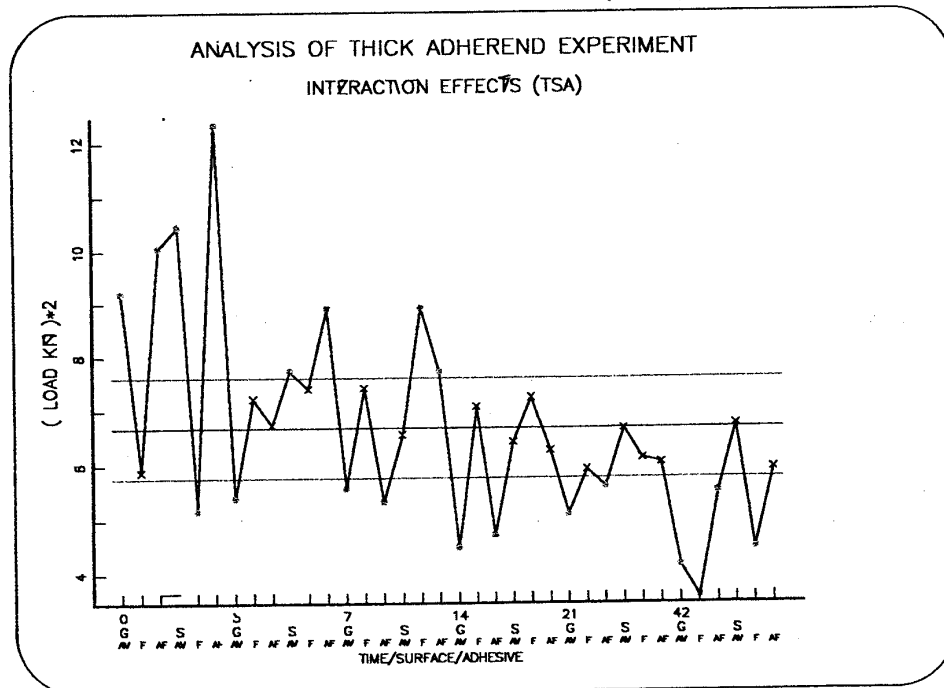
INTERACTION EFFECTS (CTA)



NOTES.

GB = GRIT BLAST ; SIL = SILANE
AV = AV119 ; F = F241 ; AF = AF126 72

APPENDIX 11

ANALYSIS OF MEANS**INTERACTION EFFECTS (CTS, CSA)****INTERACTION EFFECT (TSA)****NOTES.**

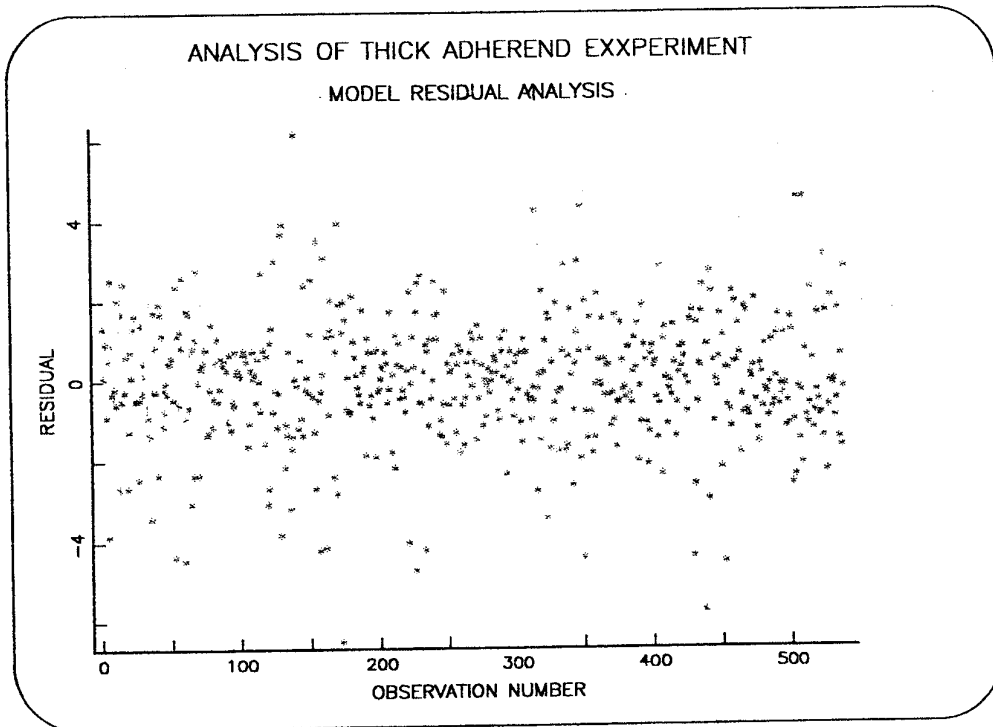
GB = GRIT BLAST ; SIL = SILANE

AV = AV119 ; F = F241 ; AF = AF126

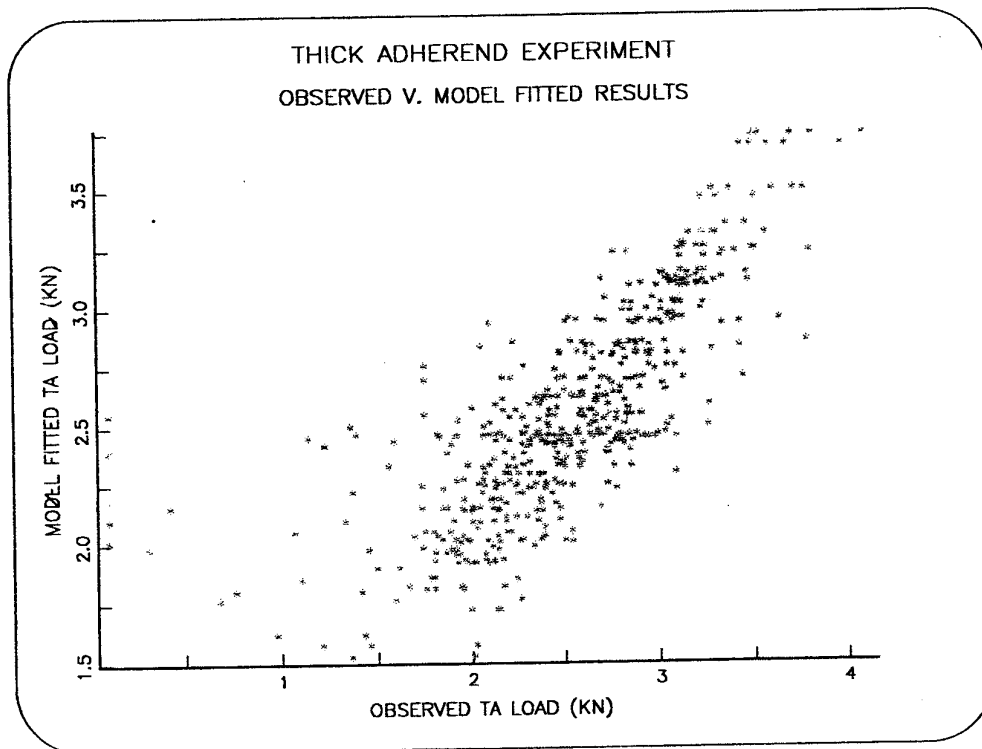
APPENDIX 12

MODEL ANALYSIS

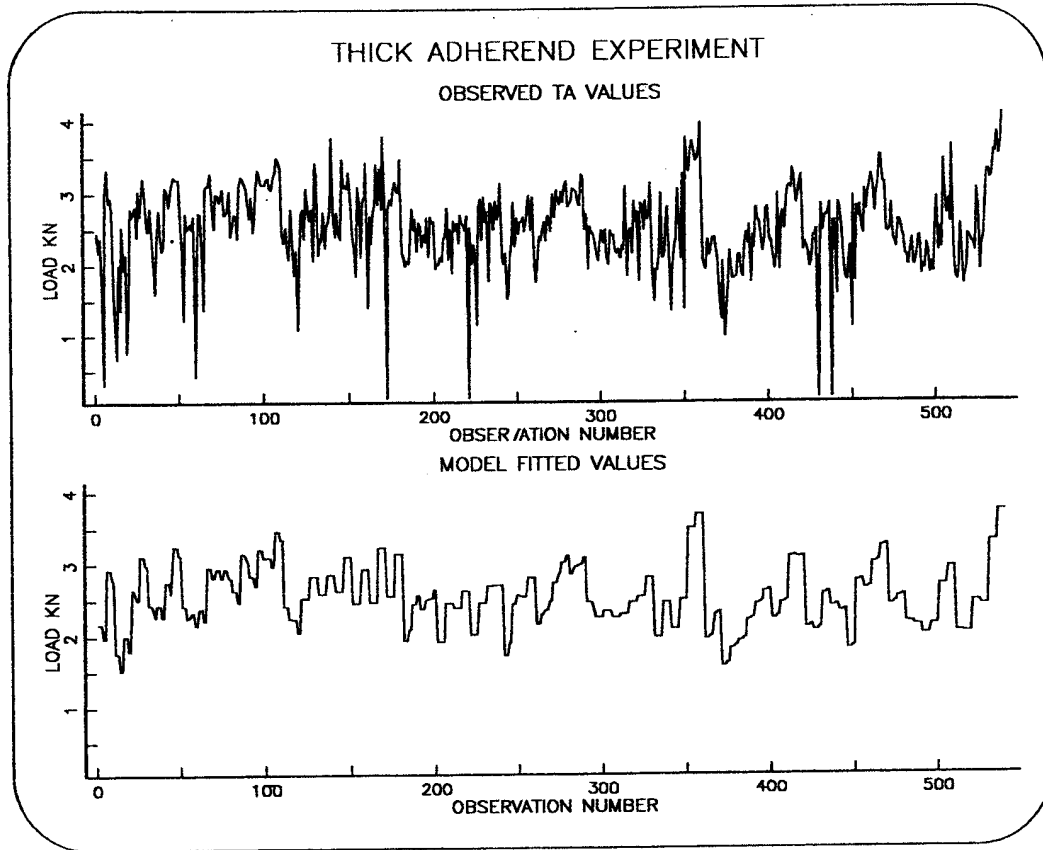
RESIDUALS AFTER MODEL FITTING



OBSERVED RESULTS v. PREDICTED FROM MODEL



APPENDIX 13

OBSERVED RESULTS v. MODEL PREDICTIONS

Case Study 4

Aluminium Vehicle Treatment Results

1. INTRODUCTION

A full factorial analysis was carried out on AVT (Aluminium Vehicle Treatment) Cure Cycle Window Data supplied by Alcan International Limited. Eight different curing conditions were assessed within the experimental programme to determine the effect of cure on the long-term performance of the joints under hot/wet conditions. A single-lap joint geometry was used for the experimental assessment. No details will be supplied as to the surface treatment, adhesive or adherend used in the programme. The objective of this case study simply being to identify the optimum processing cycle (i.e. temperature and time) based on the average strength data obtained over a period of 40 weeks.

The cure operation for joint manufacture was in two stages:

- Stage 1** Temperatures 130 °C and 150 °C / Time 5 and 15 minutes
- Stage 2** Temperatures 170 °C and 200 °C / Time 15 and 240 minutes

The average strength data is presented in Table 1. Three tests were conducted at each condition. The total number of specimens tested was 144.

Table 1 Average Strength (MPa)

Stage 1		Stage 2		Exposure Time (Weeks)					
Temp (°C)	Time (mins)	Temp (°C)	Time (mins)	0	4	8	12	20	40
130	5	170	15	23.63	21.78	22.34	22.24	21.32	20.45
130	15	170	240	23.74	20.57	19.69	20.85	19.71	17.86
130	15	200	15	24.02	21.60	19.30	20.53	20.65	19.55
*130	5	200	240	15.25	12.96	12.39	12.16	10.75	11.74
150	15	170	15	23.88	21.05	21.64	21.25	19.94	20.68
150	5	170	240	23.33	21.77	21.76	20.32	20.63	19.74
150	5	200	15	25.50	23.43	21.14	21.14	21.92	20.84
*150	15	200	240	16.69	15.65	15.47	15.03	14.51	13.95

Visual inspection shows that two sets of conditions (identified by an asterisk * in Table 1) result in poor durability. These same conditions also produce joints that are intrinsically weak (i.e. unconditioned strength) in comparison with the other six conditions. In contrast, it is not necessarily evident that a particular set of conditions result in optimum joint performance. Hence, a full factorial approach was used to quantify the main effects and interactions. Table 2 shows the factors and levels used in the full matrix of tests (Table 3).

Table 2 Factors and Levels for Full Test Matrix

Stage 1				Stage 2				Exposure Time	
A		B		C		D		E	
Temp (°C)	Level	Time (mins)	Level	Temp (°C)	Level	Time (mins)	Level	Weeks	Level
130	1	5	1	170	1	15	1	0	1
170	2	15	2	200	2	240	2	4	2
								8	3
								12	4
								20	5
								40	6

Table 3 Full Matrix of Tests

Run	Stage 1		Stage 2		Time	Failure Stress (MPa)
	A	B	C	D	E	
1	1	1	1	1	1	23.63
2	1	1	1	1	2	21.78
3	1	1	1	1	3	22.34
4	1	1	1	1	4	22.24
5	1	1	1	1	5	21.32
6	1	1	1	1	6	20.45
7	1	1	2	2	1	15.25
8	1	1	2	2	2	12.96
9	1	1	2	2	3	12.39
10	1	1	2	2	4	12.16
11	1	1	2	2	5	10.75
12	1	1	2	2	6	11.74
13	1	2	1	2	1	23.74
14	1	2	1	2	2	20.57
15	1	2	1	2	3	19.69
16	1	2	1	2	4	20.85
17	1	2	1	2	5	19.71
18	1	2	1	2	6	17.86
19	1	2	2	1	1	24.02
20	1	2	2	1	2	21.60
21	1	2	2	1	3	19.30
22	1	2	2	1	4	20.53
23	1	2	2	1	5	20.65
24	1	2	2	1	6	19.55
25	2	1	1	2	1	23.33
26	2	1	1	2	2	21.77
27	2	1	1	2	3	21.76
28	2	1	1	2	4	20.32
29	2	1	1	2	5	20.63
30	2	1	1	2	6	19.74
31	2	1	2	1	1	25.50
32	2	1	2	1	2	23.43
33	2	1	2	1	3	21.14
34	2	1	2	1	4	21.14
35	2	1	2	1	5	21.92
36	2	1	2	1	6	20.84
37	2	2	1	1	1	23.88
38	2	2	1	1	2	21.05
39	2	2	1	1	3	21.64
40	2	2	1	1	4	21.25
41	2	2	1	1	5	19.94
42	2	2	1	1	6	20.68
43	2	2	2	2	1	16.69
44	2	2	2	2	2	15.65
45	2	2	2	2	3	15.47
46	2	2	2	2	4	15.03
47	2	2	2	2	5	14.51
48	2	2	2	2	6	13.95

2. ANALYSIS OF RESULTS

2.1 COMPUTATION OF AVERAGE EFFECTS OF MAIN FACTORS

To compute the average performance of the factor A at level 1 (i.e. A_1) we add all the results in Table 3 which include factor A_1 (i.e. $A = 1$) and then divide by the number of tests. In this case, there are 24 values to be averaged. The average effect of A_1 , is therefore calculated by adding the strength results of the 24 tests. The average effects of the other factors are computed in a similar manner. Table 4 shows the average effects for all the factors for all exposure times.

Table 4 Average Effects of Main Factors

Factor	Average Value (MPa)
A_1	18.96 ± 4.02
A_2	20.05 ± 3.09
B_1	19.52 ± 4.26
B_2	19.49 ± 2.86
C_1	21.26 ± 1.43
C_2	17.76 ± 4.26
D_1	21.66 ± 1.48
D_2	17.36 ± 3.86
E_1	22.01 ± 3.55
E_2	19.85 ± 3.36
E_3	19.22 ± 3.29
E_4	19.19 ± 3.35
E_5	18.68 ± 3.68
E_6	18.10 ± 3.21

The “factorial effect” or “main effect” is the difference between the two average effects of the factor across the different levels. For example, the factorial effect of factor **C** is the difference between the average effect of C_1 and C_2 (i.e. $21.26 - 17.76 = 3.50$ MPa).

The desired outcome from the exercise is to have a higher initial strength (i.e. bigger is better) and no deterioration in strength with environmental exposure. A simpler approach is to examine the data for the unconditioned joints (i.e. 0 weeks) and for those joints conditioned for 40 weeks (see Tables 4 and 5).

Table 4 Average Effects of Main Factors on Unconditioned Joints

Factor	Average Value (MPa)
A₁	21.66 ± 3.70
A₂	22.35 ± 3.36
Difference A₂ - A₁	0.69
B₁	21.92 ± 3.94
B₂	22.08 ± 3.11
Difference B₂ - B₁	0.16
C₁	23.64 ± 0.20
C₂	20.37 ± 4.46
Difference C₂ - C₁	-3.27
D₁	24.26 ± 0.73
D₂	19.75 ± 3.82
Difference D₂ - D₁	-4.51

Table 5 Average Effects of Main Factors on Environmentally Conditioned Joints

Factor	Average Value (MPa)
A₁	17.40 ± 3.39
A₂	18.80 ± 2.83
Difference A₂ - A₁	1.40
B₁	18.19 ± 3.75
B₂	18.01 ± 2.55
Difference B₂ - B₁	-0.18
C₁	19.68 ± 1.11
C₂	16.52 ± 3.78
Difference C₂ - C₁	-3.16
D₁	20.38 ± 0.50
D₂	15.82 ± 3.15
Difference D₂ - D₁	-4.56

The information from both subsets (i.e. 0 weeks and 40 weeks exposure) and the full matrix of tests is that factor **D** has the most effect and factor **B** the least effect on joint strength. The effects of all four factors on initial joint strength and the joint strength after prolonged exposure under hot/ wet conditions are very similar.

2.2 OPTIMUM PERFORMANCE

The optimum condition for ensuring maximum initial strength is **A₂**, **B₂**, **C₁** and **D₁** with the optimum strength **S_{opt}** given by:

$$S_{opt} = T/N + (\overline{A_2} - T/N) + (\overline{B_2} - T/N) + (\overline{C_1} - T/N) + (\overline{D_1} - T/N) \quad (1)$$

where:

T = Grand total of all results = 176.04

N = Total number of results = 8

S_{opt} = Performance at optimum condition

In this case, the optimum initial strength S_{opt} predicted is 26.32 MPa. The closest result to this value was achieved with Run 31 (25.50 MPa) in Table 3.

Similarly, optimum condition for ensuring maximum durability performance is A_2 , B_2 , C_1 and D_1 with the optimum strength S_{opt} given by:

$$S_{opt} = T / N + (\overline{A_2} - T / N) + (\overline{B_2} - T / N) + (\overline{C_1} - T / N) + (\overline{D_1} - T / N) \quad (2)$$

where:

T = Grand total of all results = 144.81

N = Total number of results = 8

In this case, the optimum initial strength S_{opt} predicted is 22.75 MPa. The closest result to this value was achieved with Run 36 (i.e. 20.84 MPa) in Table 3.

In summation, the condition that most closely represents the optimum cure conditions is A_2 , B_1 , C_1 and D_1 .

2.3 INTERACTION EFFECTS

To determine whether the interaction is present between factors, a proper interpretation of the data is necessary. The average interaction effects for Stage 1 (A_1B_1 , A_1B_2 , A_2B_1 , A_2B_2) and Stage 2 (C_1D_1 , C_1D_2 , C_2D_1 and C_2D_2) are summarised in Tables 6 and 7. The tables show averages and differences between the interaction components for unconditioned (Table 6) and 40 weeks conditioning (Table 7).

Table 6 Average Effects of Interactions on Unconditioned Joints

Factor	Average Value (MPa)
A_1B_1	19.44
A_1B_2	23.88
Difference $A_1B_1 - A_1B_2$	-4.44
A_2B_1	24.42
A_2B_2	20.29
Difference $A_2B_1 - A_2B_2$	4.13
C_1D_1	23.76
C_1D_2	23.54
Difference $C_1D_1 - C_1D_2$	0.22
C_2D_1	24.76
C_2D_2	15.97
Difference $C_2D_1 - C_2D_2$	8.79

Table 7 Average Effects of Interactions on Environmentally Conditioned Joints

Factor	Average Value (MPa)
A₁B₁	16.10
A₁B₂	18.71
Difference A₁B₁ - A₁B₂	-1.61
A₂B₁	20.29
A₂B₂	17.32
Difference A₂B₁ - A₂B₂	2.97
C₁D₁	20.57
C₁D₂	18.80
Difference C₁D₁ - C₁D₂	1.77
C₂D₁	20.20
C₂D₂	12.85
Difference C₂D₁ - C₂D₂	7.35

If there was no interaction effects between **A** and **B** then **A₁B₁ - A₁B₂ = A₂B₁ - A₂B₂**. In fact, the results in Tables 6 and 7 indicate that there are interaction effects between **A** and **B**, and **C** and **D**. The differences between the average effects of the interactions are in some cases larger than the differences in the average values of the main effects (see Table 4 and 5). Hence, the optimum strength calculations should be revised to take into account the interactive components.

The revised optimum condition for ensuring maximum initial strength is **A₂, B₁, C₂ and D₁** is given by:

$$S_{opt} = T/N + (\overline{A_2B_1} - T/N) + (\overline{C_2D_1} - T/N) \quad (3)$$

where:

T = Grand total of all results = 176.04

N = Total number of results = 8

In this case, the revised optimum initial strength **S_{opt}** predicted is 27.18 MPa compared with the previous optimum value (without interaction effects) of 26.32 MPa. The revised optimum is an improvement. As previously mentioned, the closest result to this value was achieved with Run 31 (25.50 MPa) in Table 3.

The revised optimum condition for ensuring maximum initial strength is **A₂, B₁, C₁ and D₁** is given by:

$$S_{opt} = T/N + (\overline{A_2B_1} - T/N) + (\overline{C_1D_1} - T/N) \quad (4)$$

where:

T = Grand total of all results = 144.81

N = Total number of results = 8

In this case, the revised optimum initial strength **S_{opt}** predicted is 22.76 MPa compared with previous optimum prediction of 22.75 MPa. The interactions of **A x B** and **C x D** have no influence on the optimum. No further modification is required.

3. CONCLUDING REMARKS

A number of key observations can be made in regard to the data and analysis:

- The experimental programme included a set of processing conditions that closely represented the conditions for ensuring optimum initial strength and durability performance.
- Interaction effects between temperature and time at the two processing stages had only minimal effect on the overall joint performance.
- The optimum processing conditions were almost identical for both unconditioned and 40 week conditioning.

It is recommended that the interaction factors be ignored for the preliminary determination of the optimum condition. Interactions are to be included when refining the predictive model. The relative significance of the interactions can be obtained from an ANOVA (Analysis of Variance) study (see previous case studies).

ADDITIONAL READING

1. Olusanya, A., Tully, K, Mera, R. and Broughton, W.R., "A Comparison of Commercial Design of Experiment Software Programs for the Analysis of Durability Data", NPL Report CMMT(A) 98, 1998.
2. Olusanya, A., "The Use of Design of Experiments Techniques to Determine the Relative Effectiveness of Silane Coupling Agents on the Durability of Titanium Alloy Joints. A Case Study", NPL Report CMMT(A) 128, 1998.
3. Roy, R., "A Primer on the Taguchi Method", SME Society of Manufacturing Engineers, 1990.

Case Study 5

Temperature-Humidity Interaction

1. INTRODUCTION

This case study evaluates the combined effect of temperature and humidity on the durability performance of single-lap joints made from 2 mm thick Ti-6Al-4V alloy sections bonded with AF126-2 epoxy adhesive film (supplied by 3M, UK). Specimens were subjected to nine combinations of temperature and humidity for periods ranging from 0 to 17 days. Table 1 shows the factors and levels used in the full matrix of tests. Details on specimen geometry, preparation and surface treatment are given in NPL Report CMMT(A) 197 [1].

Table 1 Factors and Levels used in Full Test Matrix

Temperature (°C)	Level	Humidity (% RH)	Level	Time (days)	Level
25	1	45	1	0	1
40	2	85	2	1	2
70	3	96	3	3	3
				7	4
				17	5

2. EXPERIMENTAL RESULTS

The test results (load per unit width) obtained for the nine temperature-humidity combinations over the period of 1 to 17 days exposure are summarised in Table 2. Each average value presented in the table consists of 5 replicates. The results show a severe deterioration in strength in those joints exposed for the longest periods (7 to 17 days) under high temperature and humidity conditions (i.e. 70 °C and 96% RH).

Table 2 Effect of Elevated Temperature and Humidity on Joint Strength (N/mm)

Temperature (°C)	Relative Humidity (%)		
	45	85	96
1 day			
25 °C	368 ± 7*	281 ± 17	254 ± 17
40 °C	348 ± 28	276 ± 14	243 ± 4
70 °C	278 ± 20	273 ± 19	243 ± 7
3 days			
25 °C	368 ± 7*	270 ± 15	235 ± 10
40 °C	300 ± 6	252 ± 28	228 ± 13
70 °C	248 ± 10	240 ± 19	224 ± 21
7 days			
25 °C	368 ± 7*	276 ± 19	241 ± 13
40 °C	310 ± 17	262 ± 23	247 ± 12
70 °C	236 ± 10	244 ± 12	128 ± 19
17 days			
25 °C	368 ± 7*	187 ± 20	194 ± 15
40 °C	206 ± 15	173 ± 16	148 ± 13
70 °C	229 ± 25	195 ± 12	84 ± 9

- * Joint strength assumed constant (remains relatively unaffected for 2- 3 weeks when stored at 23 °C and 50 %RH).

3. FULL FACTORIAL MATRIX

A full factorial analysis was performed on the test results presented in Table 2. Table 3 shows the main parameters (or factors) and interaction factors, levels and corresponding failure loads.

Table 3 Full Factorial Matrix

Run	Parameter			Interactions			Failure Load
	A	B	C	A x B	A x C	B x C	(N/mm)
	Temperature	Humidity	Time				
1	1	1	1	1	1	1	368
2	1	1	2	1	1	2	368
3	1	1	3	1	1	3	368
4	1	1	4	1	1	4	368
5	1	1	5	1	1	5	368
6	1	2	1	2	1	2	368
7	1	2	2	2	2	2	281
8	1	2	3	2	2	3	270
9	1	2	4	2	4	4	276
10	1	2	5	2	5	5	187
11	1	3	1	3	1	3	368
12	1	3	2	3	2	3	254
13	1	3	3	3	3	3	235
14	1	3	4	3	4	4	241
15	1	3	5	3	5	5	194
16	2	1	1	2	2	1	368
17	2	1	2	2	2	2	348
18	2	1	3	2	3	3	300
19	2	1	4	2	4	4	310
20	2	1	5	2	5	5	206
21	2	2	1	2	2	2	368
22	2	2	2	2	2	2	276
23	2	2	3	2	3	3	252
24	2	2	4	2	4	4	262
25	2	2	5	2	5	5	173
26	2	3	1	3	2	3	368
27	2	3	2	3	2	3	243
28	2	3	3	3	3	3	228
29	2	3	4	3	4	4	247
30	2	3	5	3	5	5	148
31	3	1	1	3	3	1	368
32	3	1	2	3	3	2	278
33	3	1	3	3	3	3	248
34	3	1	4	3	4	4	236
35	3	1	5	3	5	5	229
36	3	2	1	3	3	2	368
37	3	2	2	3	3	2	273
38	3	2	3	3	3	3	240
39	3	2	4	3	4	4	244
40	3	2	5	3	5	5	195
41	3	3	1	3	3	3	368
42	3	3	2	3	3	3	243
43	3	3	3	3	3	3	224
44	3	3	4	3	4	4	128
45	3	3	5	3	5	5	84

The average effects of the main factors of temperature, relative humidity and exposure time on joint strength are shown in Figures 1 to 3. Figure 4 shows the average effect of the interaction between temperature and humidity. The full factorial matrix was analysed

using Microsoft Excel Version 7 spreadsheet software. The software enabled the determination of regression fits to the data; as shown by the equations in Figures 1 to 3. The process for determination of the main factor effects and interaction effects are identical to those employed in the previous case study. It was clearly evident from the analysis that there was a synergistic effect between temperature and humidity, although this was difficult to express as an empirical formulation (Figure 4).

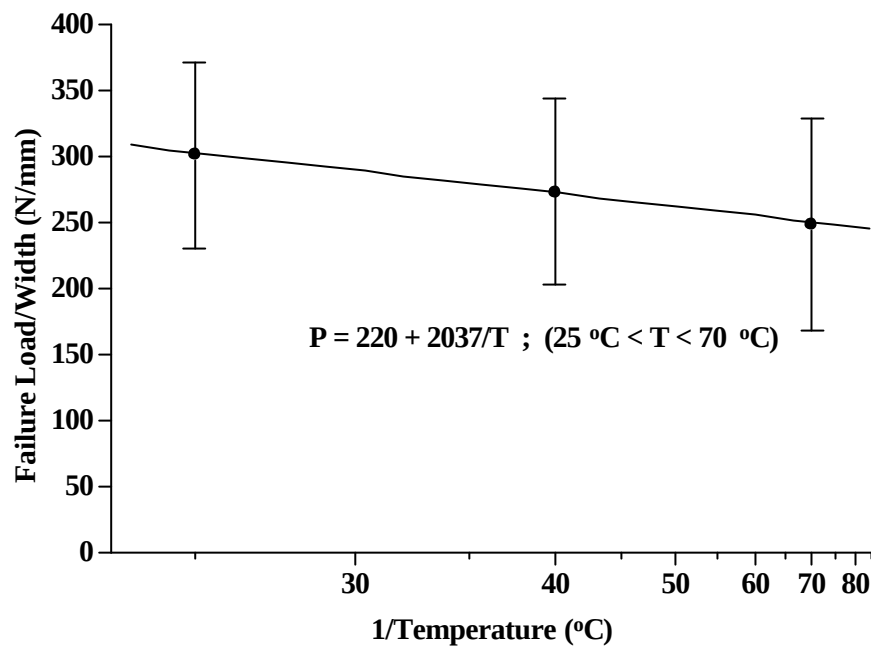


Figure 1 Effect of temperature on failure load for 6Al-4V-Ti alloy / AF126 single-lap joints.

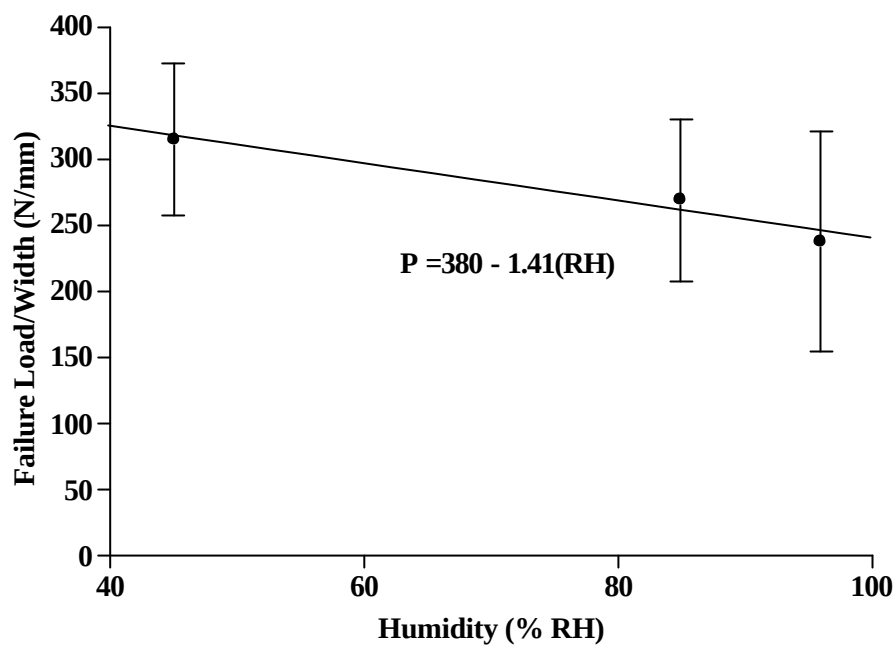


Figure 2 Effect of humidity on failure load for 6Al-4V-Ti alloy / AF126 single-lap joints.

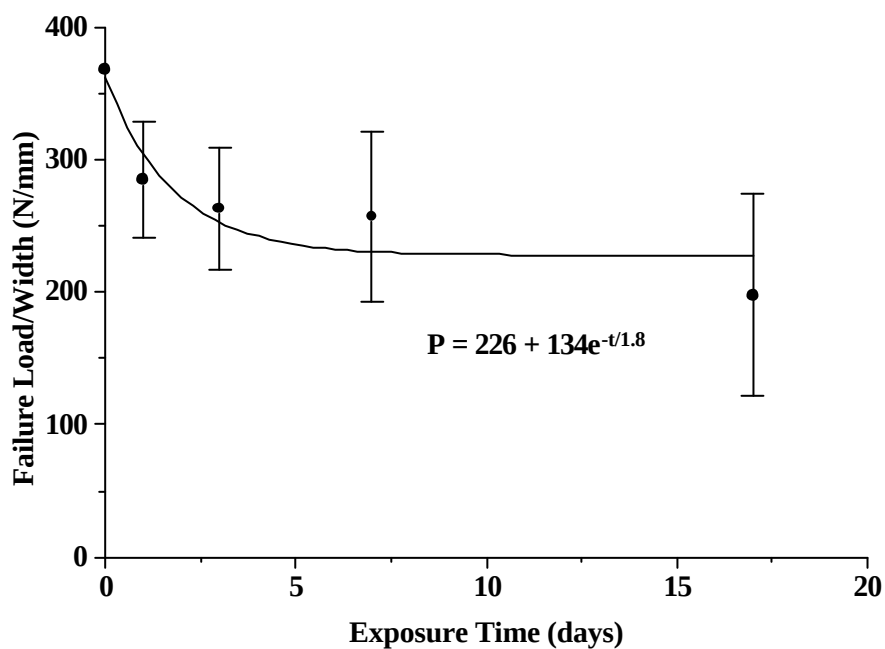


Figure 3 Effect of time on failure load for 6Al-4V-Ti alloy / AF126 single-lap joints.

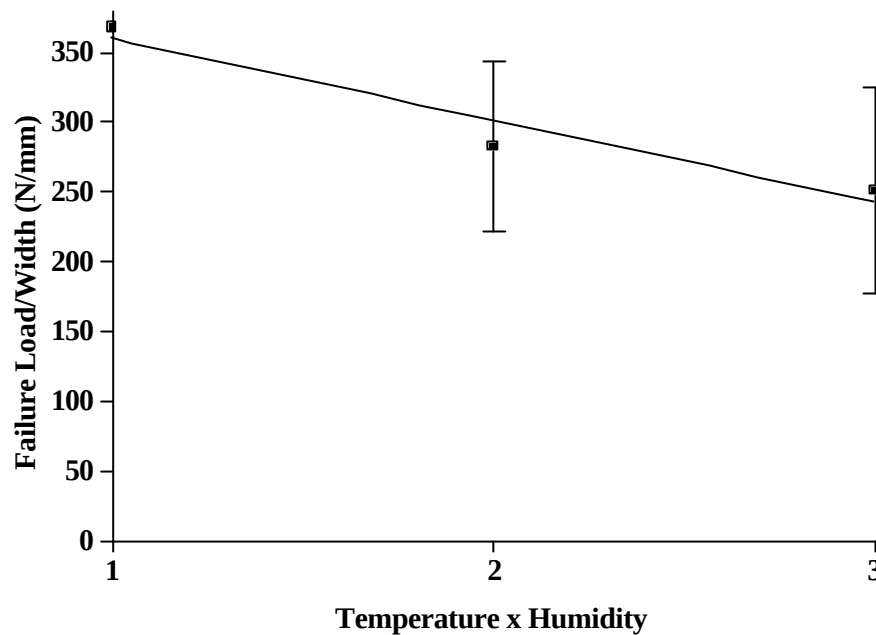


Figure 4 Interaction between temperature and humidity.

Figures 1 to 3 include empirical equations relating joint strength to the three main factors of Temperature (**A**), Relative Humidity (**B**) and Exposure Time (**C**). The failure strength of the joint for any combination of the three main factors can be calculated from the three constitutive equations and the grand average performance (GAP) value obtained from the full factorial experiment. The equation for calculating the joint strength is given below:

$$\text{Failure strength} = \text{GAP} + (\text{Eqn (A)} - \text{GAP}) + (\text{Eqn (B)} - \text{GAP}) + (\text{Eqn (C)} - \text{GAP}) \quad (1)$$

Note. GAP value was calculated to be 274 N/mm.

The above empirical relationship tends to underestimate the joint strength for most test conditions. At elevated temperature and relative humidity the reverse occurs with the predicted values being higher than those measured. Table 4 shows a comparison between measured failure load and predictive model for data obtained after 7 days exposure. Whilst the constitutive equations relating joint strength to temperature and relative humidity provide reasonable fits to the data (Figures 1 and 2), the constitutive equation relating joint strength to exposure time (see Figure 3) is a much poorer fit. The poor correlation between this equation and measured strength values adversely affects the reliability of Equation (1) in predicting joint strength. Although the inclusion of interaction effects in Equation (1) may improve the degree of correlation between predictive model and measured data, it would also significantly increase the degree of complexity of the analysis.

Table 4 Comparison of Predicted and Measured Joint Strength (N/mm) After 7 Days

Temperature (°C)	Relative Humidity (%)		
	45	85	96
25 °C			
Measured	368 ± 7	276 ± 19	241 ± 13
Predicted	299	242	227
40 °C			
Measured	310 ± 17	262 ± 23	247 ± 12
Predicted	268	212	196
60 °C			
Measured	236 ± 10	244 ± 12	128 ± 19
Predicted	251	195	179

4. CONCLUDING REMARKS

The results presented in this section show that for this particular system and joint configuration simple algebraic relationships can be used to estimate the effects of temperature and humidity, and their interactions, on joint strength. The equations, however are empirical, and therefore not related to any physical mechanism. The overall equation relating to the main factors is only as good as the constitutive relationships incorporated. Any deficiencies in these relationships will result in poor correlation between the predictive model and the measured data. In addition, the large uncertainty often associated with durability data can often mask interaction effects and adversely contribute to the level of agreement between predicted and measured data.

REFERENCES

1. Broughton, W.R. and Mera, R.D., "Environmental Degradation of Adhesive Joints. Accelerated Testing", NPL Report CMMT(A) 197, 1999.