#### THE SPRING RESEARCH AND MANUFACTURERS' ASSOCIATION

# HYDROGEN EMBRITTLEMENT OF HARDENED AND TEMPERED CARBON SPRING STEEL STRIP ELECTROPLATED WITH ZINC

First Progress Report

THE EFFECTS OF HYDROGEN CONTENT,

ZINC THICKNESS AND BAKING TIME

UPON HYDROGEN EMBRITTLEMENT

Report No. 345

by

L.F. Reynolds, M.Sc. Tech., C.Eng., M.I.M.

Report No. 345

#### THE EFFECTS OF HYDROGEN CONTENT, ZINC THICKNESS AND BAKING TIME UPON HYDROGEN EMBRITTLEMENT

#### SUMMARY

The relationship between the hydrogen content and the degree of embrittlement of carbon spring steel strip was assessed using a slow bend test technique, after plating with zinc to nominal deposits corresponding to Zn2 (Class C) and Zn10 (Class A) respectively of BS 1706.1960. The efficacy of de-embrittlement treatments was assessed after baking the plated strips for times of 2 hours and 18 hours at 190/200°C.

The fracture surfaces of selected bend test samples were subsquently examined on the Scanning Electron Microscope.

It was concluded that the bulk hydrogen content of the steel could not be related to the degree of embrittlement. The hydrogen content of the strip, measured with the zinc in situ, did show a significant direct correlation with the zinc thickness.

The degree of embrittlement was not significantly affected by the thickness of the zinc depost. There was some evidence to show that the strips with Zn2 deposits responded more favourably than those with Zn10 deposits to the appropriate de-embrittlement baking treatments, however.

A 2 hour baking treatment did not significantly improve the ductility of either the Zn2 or the Zn10 plated strips.

Baking for 18 hours significantly improved the ductility of the Zn 2 strips, but did not improve that of the Zn10 plated material.

The final ductility of the plated strips after baking for 18 hours always remained significantly lower than that of the unplated strips.

There was evidence to suggest that a number of the strips still exhibited severe hydrogen embrittlement despite baking for 18 hours at 190/200°C.

The work has shown that suitable fractographic analysis, using the Scanning Electron Microscope, can reveal clear evidence of hydrogen embrittlement.

#### ALL RIGHTS RESERVED

The information contained in this report is confidential and must not be published, circulated or referred to outside the Association without prior permission.

#### CONTENTS-

			Page No
1.	INTF	RODUCTION	1
2.	EXPE	ERIMENTAL PROCEDURE	2
	2.1	Material and Specimen Preparation	2
	2.2	Cleaning and Electroplating	2
	2.3	De-embrittlement Baking	3
	2.4	Slow Bend Tests	3
	2.5	Zinc Thickness	4
	2.6	Hydrogen Content	4
3.	RESU	ULTS AND DISCUSSION	5
	3.1	Effect of Hydrogen Content on Bend Angles	5
	3.2	Preliminary Statistical Analysis of Bend Angle	6
		Results for Zinc Plated and Baked Samples	
	3.3	Effect of Pre-Cleaning and Zinc Thickness	7
		on Bend Angles	
	3.4	Effect of Baking Time on the Bend Angles	8
		of Zinc Plated Strips	
	3.5	Examination of Fractures on Scanning Electron	9
		Microscope.	
4.	CONC	CLUSIONS	10
5.	RECO	OMMENDATIONS FOR FURTHER WORK	11
6.	REFE	ERENCES	12
7.	TABI	LES :	
	I	TREATMENTS USED FOR ASSESSMENT OF HYDROGEN EMBRITTLEMENT RESULTING FROM ZINC ELECTRO-PLATING OF CARBON SPRING STEEL STRIP.	
	II	SLOW BEND TEST RESULTS FOR CARBON SPRING STEEL STRIP	
	III	ZINC THICKNESS OF ELECTROPLATED CARBON SPRING STEEL STRIP	

- IV HYDROGEN ANALYSES OF CARBON SPRING STEEL STRIP
- V RESULTS OF 3-WAY ANOVA FOR SLOW BEND TEST PROPERTIES OF ZN2 AND ZN10 PLATED CARBON SPRING STEEL STRIPS BAKED FOR 2 HOURS AND 18 HOURS AT 190/200°C.
- VI EFFECTS OF PRE-CLEANING AND ZINC PLATING/BAKING TREATMENTS ON THE SLOW BEND TEST PROPERTIES OF CARBON SPRING STEEL STRIP.
- VII EFFECT OF BAKING TREATMENTS UPON SLOW BEND TEST PROPERTIES OF Zn2 PLATED CARBON SPRING STEEL STRIPS
- VIII EFFECT OF BAKING TREATMENTS UPON SLOW BEND TEST PROPERTIES OF ZN10 PLATED CARBON SPRING STEEL STRIPS
- IX CLASSIFICATION OF SLOW BEND TEST RESULTS FOR ZN2 PLATED CARBON SPRING STEEL STRIPS
- X CLASSIFICATION OF SLOW BEND TEST RESULTS FOR ZN10 PLATED CARBON SPRING STEEL STRIPS

#### 8. FIGURES

- 1. Angle at Fracture during Slow Bend Test plotted against Hydrogen Content of Zinc Plated Carbon Spring Steel Strip.
- 2. Angle at Fracture during Slow Bend Test plotted against Hydrogen Content of Untreated, Acid Cleaned, and Zinc Plated Carbon Spring Steel Strips from which the Zinc has been removed.
- Total Hydrogen Content of Zinc Plated Carbon Spring Steel Strip plotted as a function of Zinc Thickness.
- 4. Frequency Distribution of Bend Angles for Untreated, Acid Cleaned and Zn2 Plated Carbon Spring Steel Strips before and after baking at 190/200°C.
- 5. Frequency Distribution of Bend Angles for Untreated, Acid Cleaned and ZnlO Plated Carbon Spring Steel Strips before and after baking at 190/200°C.
- 6. Scanning Electron Fractograph of Unplated Strip Showing Ductile Dimpled Fracture (Sample A5, Bend Angle = 1100).
- 7. Scanning Electron Fractograph of Zinc Plated Strip Showing Typical Intergranular Fracture Due to Hydrogen Embrittlement (Sample C9, Bend Angle = 550).

# THE EFFECTS OF HYDROGEN CONTENT, ZINC THICKNESS AND BAKING TIME UPON HYDROGEN EMBRITTLEMENT

by

L. F. Reynolds, M.Sc. Tech., C. Eng., M.I.M.

Report No. 345

#### 1. INTRODUCTION AND OBJECTIVE

Modern developments in the techniques available for the determination of hydrogen in metals have made it possible to measure the total hydrogen content of steel with greater precision that that obtainable using methods of hot extraction from the solid metal under vacuum.

The present work was designed to measure the hydrogen content of carbon spring steel strip both before and after the application of selected zinc plating and baking treatments. Such measurements could then be examined in association with the ductility of the strip as determined by a slow bend test.

It is known that commercial barrel plating produces a range of zinc thickness, and the effects of two widely disparate levels of mean zinc thickness upon the hydrogen content and degree of embrittlement were therefore investigated.

Previous work at SRAMA had indicated that baking for 2 hours at  $190/200^{\circ}$ C, after zinc plating, was potentially as effective as a 16 - 24 hour bake in reducing the harmful effects of hydrogen embrittlement upon hardened and tempered carbon spring steels (1).

In view of this finding, the present work was therefore designed to examine more closely the effects of baking time upon the ductility of the zinc plated strip. It was further intended to characterise the fracture surfaces of ductile and hydrogen embrittled strips, broken during slow bend testing, by means of the SRAMA Scanning Electron Microscope.

In the event, the extent of this scanning electron fractography had to be restricted due to lack of time and a complete fracture characterisation therefore remains to be completed in the future.

#### 2. EXPERIMENTAL PROCEDURE

#### 2.1 Material and Specimen Preparation

Hardened and tempered carbon spring steel polished strip was obtained as coil to BS 1449. Part 1. 1972 with a hardness of 540 - 560 Hc and "as sheared" dimensions of 12 mm width x 0.8 mm thickness. Examination of the strip revealed the material to possess a hardness of 551 - 553 Hv20, the chemical composition being as follows:-

The strip was cut into test pieces each of 90 mm length, after which both longitudinal edges were equally surface ground by means of Elliot 618 precision tool room grinder incorporating a magnetic table, to give a final specimen width of 11.4 mm, thus reducing the effects associated with the original sheared edge of the strips.

#### 2.2 Cleaning and Electroplating

The appropriate strips were commercially acid cleaned and zinc electroplated by a member firm, so as to produce results which would be generally applicable to the spring industry. The strips were cleaned by batch immersion in a commercial solution of inhibited acid.

Electroplating was carried out at a current density of approximately 3A/dm<sup>2</sup> in a low cyanide barrel plating bath, the approximate composition of which was as follows:-

Zinc 12g/1
Total Sodium Cyanide 20g/1
Sodium Hydroxide 75g/1

One barrel load of strips was zinc plated to an average thickness of 0.038 mm, as recommended for BS Zn10, Class A of BS 1706.1960, whilst an equal batch was similarly plated to an average thickness of 0.009 mm, corresponding to BS Zn2, Class C of the same specification.

#### 2.3 De-embrittlement Baking

Baking was carried out by a member firm at a temperature of  $190 - 200^{\circ}$ C within 1 hour of completion of electroplating.

One batch of each zinc thickness was not baked; similar batches of each zinc thickness were baked for 2 and 18 hours.

#### 2.4 Slow Bend Tests

The angle at which the strips broke when bent around a 4.53 mm diameter mandrel at a rate of 0.6 degrees/sec. was used as a measure of ductility. Equal batches of 25 strips/batch were tested using the slow bend test machine described in an earlier report (2).

One batch of strips was tested in the "untreated" condition, i.e. prepared by surface grinding but neither cleaned nor plated to give a measure of the original ductility of the strip.

A further batch of strips was tested after acid cleaning, to assess the effect of this process upon embrittlement. The remaining zinc plated strips, after baking if appropriate, were tested in equal sized batches, to the experimental design shown in Table I whilst the test results are given in Table II. The fracture surfaces were removed from selected samples of the broken strip as soon as possible after testing, and were individually stored in dessicant for future examination on the Scanning Electron Microscope.

#### 2.5 Zinc Thickness

Measurements of the average zinc thickness were made using the techniques described in Appendix B of BS 1706. 1960.

Sections of strip were taken from the broken samples obtained as a result of the bend tests. After appropriate dimensional measurement, the sections were weighed, after which the zinc was removed using an acidified solution of antimony trichloride. The strips were re-weighted after removal of the antimony deposit and were dried, the thickness of zinc being subsequently calculated from the relationship:

Average zinc thickness (mm) =  $\frac{W}{A}$  x 141

Where W = Weight of Zinc removed, gm.,
A = Area of zinc coated surface, mm<sup>2</sup>.

The results of the determinations are shown in Table III.

#### 2.6 Hydrogen Content

The hydrogen content of selected strips was measured using a Balzer Halograph EAH220 vacuum fusion instrument. Three strips were selected for hydrogen measurement from each of the eight batches shown in Table I, the selection being in each case representative of the maximum, medium and minimum values of bend angle obtained during bend testing. In the case of batches C - J, which had all been zinc plated, hydrogen determinations were made both with the zinc present, and on different sections of the same strip after the zinc had been removed using the technique described in Section 2.5 of the report.

The results of the hydrogen analyses are presented in Table IV.

#### 3. RESULTS AND DISCUSSION

#### 3.1 Effect of Hydrogen Content on Bend Angle

The results of the hydrogen analyses are shown plotted against bend angle for the zinc plated strips in Fig. 1 and for the equivalent strips from which the zinc had been removed in Fig. 2.

It is apparent that there was no correlation, in either case, between the hydrogen content of the strip and the ductility as represented by the bend angle.

These values of hydrogen content were averages obtained throughout the bulk of specimen, however. It should be appreciated that the hydrogen content of the steel immediately adjacent to and just below the zinc plated surface could have been very significantly higher than that quoted for the bulk of the steel itself. The regions of highest hydrogen content at the surface would therefore coincide with the regions of highest tensile bending stress leading to the development of conditions favourable to hydrogen embrittlement.

The hydrogen content of the zinc plated strip was at least an order of magnitude higher than that for the samples from which the ainc had been removed, suggesting that a significant proportion of the total hydrogen was contained within the zinc layer rather than in the steel substrate.

The plot of Fig. 3 shows the hydrogen content as a function of the zinc thickness for the present work, and incorporates the results of earlier work carried out at SRAMA on behalf of a member firm (3). The relationship indicates that the total hydrogen content of the plated strips tended to increase directly as the zinc thickness increased. Such a relationship suggests that a substantial fraction of the total hydrogen detected in the zinc plated strip was associated with the zinc plate itself.

## 3.2 <u>Preliminary Statistical Analysis of Bend Angle Results</u> for Zinc Plated and Baked Samples

A three way Analysis of Variance (ANOVA) technique was used to investigate the main sources of variation present in all the raw bend angle data.

The final simplified results of this analysis are shown in Table V. The results effectively indicated that the baking time was the only single factor directly affecting the bend angle to any significant extent. A relatively significant contribution to the overall variation of the results was provided by the Zinc Thickness X Baking Time interaction, however.

Estimates of the relative contributions of these effects to the overall variance of the bend test results yielded the following results:

Source of Variation	Variance Contribution
Baking time	3.1
Zinc Thickness x Baking Time	20.5
Residual (Error)	76.4
Total	100.0

The relatively large contribution of the variance of the Zinc Thickness x Baking Time interaction suggests that some combinations of baking time and zinc thickness gave bend angles with unexpectedly high or unexpectedly low results.

Frequency distributions were therefore plotted for the bend angle results obtained for the Zn2 and Zn10 plated strips, and these are shown in Figs. 4 and 5 respectively.

Two main points of interest emerge from consideration of the ANOVA results and the frequency distributions.

- 1. The Zn2 strips appeared to be more effectively de-embrittled than the Zn10 strips by the 18 hour baking treatment. (This effect would account for the large Zinc Thickness x Baking Time interaction).
- The bend test results did not generally conform to 2. a Normal distribution indicating that the variances of the bend test results could not be assumed to be independent estimates of the same population In consequence, Students 't' test would variance. not be the most discriminating technique for assessing the significance of the difference in mean values of bend angle between appropriate batch samples. The Welch-Test was therefore selected for further statistical analysis of the bend test results, since this technique is completely independent of any knowledge of population variances, other than assuming that the information is contained within the raw data obtained during the course of the experiments.

Further details of the Welch Test and its application can be found in the literature  $^{(4)}$ .

# 3.3 Effect of Pre-cleaning and Zinc Thickness on Bend Angles

The results of the statistical analyses are shown in Table VI. The commercial pre-cleaning treatment in inhibited acid produced a statistically significant reduction of  $3^{\circ}$  in the bend angle.

Such a reduction in ductility would not be likely to prove significant in practice, however, especially since examination of the frequency distribution (Fig. 4) shows that the low angles characteristic of severe embrittlement were completely absent.

In general, the degree of embrittlement produced by zinc plating was not significantly influenced by the thickness of the zinc deposit.

# 3.4 Effect of Baking Time on the Bend Angles of Zinc Plated Strips

The results of the statistical analyses for the Zn2 and Zn10 plated strips are shown in Table VII and VIII respectively.

The analyses indicate that, in the absence of de-embrittlement, zinc plating always resulted in a significantly reduced ductility.

There was no improvement in the ductility of either the Zn2 or the ZN10 plated strips after baking for 2 hours, nor of the Zn10 plated strips after baking for 18 hours. The Zn2 plated strips showed a significant improvement in ductility after baking for 18 hours, however.

These findings do not conform entirely to those of earlier work at SRAMA into the effects of baking time upon the ductility of similar zinc plated carbon spring steel strip, where it was found that a 2 hour bake at 190/200°C was as effective as a 24 hour bake in alleviating hydrogen embrittlement (1).

The present results are in agreement with those of the earlier work, however, in that the ductility of the de-embrittled strip always remained significantly lower than that of the unplated strip.

To throw further light upon these inter-relationships, the raw bend test data was classified in terms of bend angle as follows:

Description of Behaviour in Slow Bend Test	Bend Angle at Fracture
Not embrittled	>109 <sup>O</sup>
Slightly/Moderately Embrittled	80 <sup>0</sup> -109 <sup>0</sup>
Severely Embrittled	<80 <sup>0</sup>

The proportions of the appropriate data contained within this classification are shown in Tables IX and X for the Zn2 and Zn10 plated strips respectively.

Examination of the data thus classified confirms that only the Zn2 plated strips showed any improvement in ductility after baking for 18 hours.

The results also indicate that a proportion of all the zinc plated strips still exhibited significantly impaired ductility even after baking for 18 hours at  $190/200^{\circ}$ C. This finding may require further investigation in future work.

#### 3.5 Examination of Fractures on Scanning Electron Microscope

A full examination of the appropriate fracture surfaces, obtained during slow bend testing of the strips, was not possible during this work due to the limitations imposed by the time available. A selective examination was therefore undertaken of typical fractures representative of the untreated strip (Batch A) and the Zn2 "as plated" strip (Batch C).

Scanning electron fractographs typical of those observed for Sample A5 (Bend Angle  $110^{\circ}$ ) and Sample C9 (Bend Angle  $55^{\circ}$ ) are shown in Figs. 6 and 7 respectively.

The fracture surface of Sample A5 displayed the dimpled appearance characteristic of a ductile material. Sample C9, by contrast, displayed fracture areas typical of the brittle intergranular failure mode often associated with the hydrogen embrittlement of high tensile steel, especially at those regions of the strip immediately adjacent to the surface which was stressed in tension during bending.

These observations corroborate the findings of recent work by Hirth et al, who investigated the effect of hydrogen induced embrittlement upon the fracture mode of high tensile alloy steels used in screw fastener applications, where the principal stresses would be applied in uniaxial tension (5,6)

One example of a spring steel embrittled by hydrogen was provided, however, in the form of a zinc electroplated 685A55 alloy (Chromium-Silicon,  $\sim$  En48A) at a tensile strength of 2200 N/mm<sup>2</sup> which fractured in bending upon the first load application, despite a post plating bake of 190°C for 1 hour.

Typical steels examined experimentally included 527A60 (Carbon/Chromium,  $\sim$  En48) and 708A42 (Chromium/Molybdenum,  $\sim$  En19A), hardened and tempered to tensile strengths within the range 600 - 2200 N/mm<sup>2</sup>.

These authors concluded that the steels which they examined exhibited a progressively increasing sensitivity to the effects of hydrogen embrittlement as the tensile strength. increased above ∿ 1000 N/mm<sup>2</sup>. Their work established that the techniques of fractographic analysis on the Scanning Electron Microscope were suitable for identifying hydrogen induced brittle failures in these steels, and that such analysis could indicate the presence of hydrogen embrittlement even when the appropriate tensile mechanical properties of the steels were unaffected. Furthermore, examination on the scanning electron microscope of a hydrogen embrittled 527A60 steel also revealed evidence of microcracks existing at the embrittled surface of the steel (6). feature of surface microcracks may require some attention in future investigations into the hydrogen embrittlement of spring steels.

#### 4. CONCLUSIONS

- 1. The hydrogen content of the bulk strip, either with the zinc in situ or with the plating removed, could not be correlated with the level of ductility as represented by the angle of bend at fracture during slow bend testing.
- 2. A significant correlation existed between the total hydrogen content of the zinc plated strip and the thickness of the zinc plate, suggesting that a significant proportion of the

- hydrogen was located in the zinc plate rather than in the steel strip.
- 3. Electroplating with zinc always resulted in a significant reduction in the ductility of the "as plated" strip. The degree of embrittlement was not significantly affected by the thickness of the zinc plate, however.
- 4. Irrespective of the zinc thickness, baking at 190/200°C, for 2 hours did not produce any significant improvement in the ductility of the plated strip.
- 5. The Zn2 (Class C) plated strip exhibited a significant improvement in ductility after baking at 190/200°C, for 18 hours.
- 6. The Zn10 (Class A) plated strip did not exhibit any significant improvement in ductility after baking at 190/200°C for 18 hours.
- 7. Irrespective of zinc thickness, a number of the plated strips still exhibited severe hydrogen embrittlement after baking at 190/200°C for 18 hours.
- 8. Irrespective of treatment, the ductility of the zinc plated and baked strips always remained significantly lower than that exhibited by the unplated steel strip.
- 9. The techniques of fractographic analysis made possible by the Scanning Electron Microscope are particularly suitable for identifying hydrogen induced brittle fractures in high tensile spring steels.

#### 5. RECOMMENDATIONS FOR FURTHER WORK

1. The results of the present work suggest that baking for 2 hours at 190/200°C will not significantly improve the ductility of zinc plated carbon spring steel strips. This is in contrast to the results of earlier work at SRAMA, which suggested that a 2 hour bake was as effective as a 24 hour bake in improving the ductility of a similar zinc plated spring steel. Further work is required to establish the reasons for this apparent disparity of experimental findings.

2. Further work should be carried out to throw further light upon the finding that a number of the zinc plated strips still exhibited severe embrittlement even after baking at 190/200°C for 18 hours.

This aspect of the work should be pursued with some urgency, since the finding is likely to be of particular significance for the service performance of carbon and low alloy steel springs which have been zinc plated for corrosion protection.

3. A full fractographic analysis of the appropriate fractures should be undertaken on the Scanning Electron Microscope, to establish more fully the character of fractures observed in carbon spring steels which have experienced hydrogen embrittlement.

The steel surface of the plated strip should also be examined for evidence of microcracks developed as a result of hydrogen embrittlement.

#### 6. REFERENCES

- Desforges, J., "An Investigation into the Removal of Hydrogen Embrittlement by Baking". SRAMA Report No. 292, April, 1978.
- Mee, J.E., "The Hydrogen Embrittlement of Electroplated Cold Worked Steels". SRAMA Report No. 150, July, 1964.
- 3. Partridge, A.R., Lewis Spring Products Limited, Private Communication.
- 4. Mack, C., "Essentials of Statistics for Scientists and Technologists". Plenum Press, 1967.
- 5. Hirth, F. W. and Speckhardt, H., Wire, 29, 1979 (3) pp 95-104
- 6. Ibid, Wire, 29 1979, (4), pp 168-173.

TABLE I TREATMENTS USED FOR ASSESSMENT OF HYDROGEN EMBRITTLEMENT RESULTING FROM ZINC ELECTROPLATING OF CARBON SPRING STEEL STRIP.

BATCH IDENTIFICATION	BATCH TREATMENT
A	UNTREATED
В	ACID CLEANED
С	AS PLATED, BS 1706, Zn2
D	AS C, + 2 HOURS DE-EMBRITTLEMENT 190/200°C
Е	AS C, + 18 HOURS DE-EMBRITTLEMENT 190/200°C
Н	AS PLATED, BS 1706. Zn10
I	AS H, + 2 HOURS DE-EMBRITTLEMENT 190/200°C
J	AS H, + 18 HOURS DE-EMBRITTLEMENT 190/200°C

TABLE II SLOW BEND TEST RESULTS FOR CARBON SPRING STEEL STRIP

ватсн	NUMBER OF TESTS	MEAN BEND ANGLE θ (degrees)	STANDARD DEVIATION
A	25	116.4	2.7
В	25	113.1	3.2
С	25	90.2	15.4
D	25	86.7	9.5
E	25	99.9	12.7
Н	25	89.4	10.7
I	25	92.2	9.2
J	25	93.0	13.2

TABLE III ZINC THICKNESS OF ELECTROPLATED CARBON SPRING STEEL STRIP

PARAMETER MEASURED	BATCH IDENTIFICATION						
MEASURED	С	D	Е	Н	I	J	
Mean Zinc thickness μm	6.0	6.1	5.4	21.4	14.7	16.7	
Standard Deviation, µm	1.0	າ.8	0.7	1.4	1.4	3.6	
No. of strips tested.	25	25	25	25	21	13	

HYDROGEN ANALYSES OF CARBON SPRING STEEL STRIP

TABLE IV

<del></del>						
		ing at 00°c	18 hrs	2.6	0.1	3
pedd.	Zn 10	Bak time 190/2	2 hrs	4.7	6.0	3
and Stri		As Plated		1.3	0.1	æ
lated		ing at 00°C	18 hrs	2.0	9.0	٣
Zinc P	Zn 2	Bak time 190/2	2 hrs	2.8	1.1	3
		As Plated		2.3	0.3	٣
		ing at 000c	18 hrs	52.1	12.6	ю
	Zn 10	Baki time 190/20	2 hrs	44.1	3.5	3
Zinc Plated		As Plated		47.1	4.9	3
		ing at 00°C	18 hrs	16.8	1.5	Э
	Zn 2	Bak time 190/2	2 hrs	14.5	3.5	3
		As Plated		20.7	5.0	က
<del></del>				1.7	0.4	К
			1.1	0.3	ю	
Parameter Measured R		Mean Hydrogen Content P.p.m.	Standard Deviation, P.p.m.	No. of strips analysed		
	Zinc Plated Zinc Plated and Stripped	Zinc Plated Zinc Plated and Stri	Received cleaned As Asid Received shaped $\frac{2 \ln c}{190/200^{\circ}c}$ Plated $\frac{3 \ln c}{190/200^{\circ}c}$ Plated	As Acid Received cleaned As Plated and Strip Received cleaned baking blated $a$		As Acide and Strip Received cleaned baking time at time

RESULTS OF 3 WAY ANOVA FOR SLOW BEND TEST PROPERTIES OF Zn2 AND Zn10 PLATED CARBON SPRING STREE STRIPS BAKED FOR 2 HOURS AND 18 HOURS AT 190/2000C TABLE V

SOURCE OF VARIATION	CORRECTED SUM OF SQUARES	DEGREES OF FREEDOM	MEAN	VARIANCE RATIO	SIGNIFICANCE AND COMPONENTS OF VARIANCE ESTIMATED BY MEAN SQUARE
Between Times (Columns)	1218.01	1	1218.01	9.71	Significant at 99.5%, De-embrittlement time has significant influence upon bend angle (b.r $s_c^2 + rs_{b.c.}^2 + s_o^2$ ).
Interaction Zn x Time (Block & Column)	967.21	1	967.21	7.71	Significant at 99%. Bend Angle varies significantly with some Zn thickness/de-embrittlement time combinations. (r. S <sub>b.c.</sub> + S <sub>o</sub> ).
Residual (error)	12169.53	26	125.46		s <sub>o</sub> s
Total	14354.75	66			

EFFECTS OF PRE-CLEANING AND ZINC PLATING/BAKING TREATMENTS ON THE SLOW BEND TEST PROPERTIES OF CARBON SPRING STEEL STRIP. TABLE VI

	_			
CONCLUSIONS	Pre-cleaning with inhibited acid solution caused a significant reduction in ductility	Zinc thickness had no significant effect on the reduction of ductility of the "as plated" strips.	After 2 hour bake the ductility of the 2n10 strip may have been significantly better than that of the Zn2 strip.	After 18 hour bake, there was no significant difference in the ductility of the Zn2 strip and the Zn10 strip.
SIGNIFICANCE	866<	%06×	>958	<958
DEGREES OF FREEDOM $(V_1=V_2)$	24	24	24	24
Ч	0.42	0.67	0.52	0.48
X	3.94	0.21	2.08	1.88
BATCHES COMPARED	8 + A	H +	I + Q	Ю + Ю

EFFECT OF BAKING TREATMENTS UPON SLOW BEND TEST PROPERTIES OF 2n2 PLATED CARBON SPRING STEEL STRIPS TABLE VII

,					
CONCLUSIONS	<pre>Zn (2) plating caused by a highly significant reduction in ductility.</pre>	2 hour bake did not significantly improve ductility.	18 hour bake significantly improved ductility.	After baking for up to 18 hours the ductility of the plated strips was significantly lower	than that of the unplated strips.
SIGNIFICANCE	866<	<b>\$06&gt;</b>	>98%	\$66<	866<
DEGREES OF FREEDOM $(V_1=V_2)$	24	24	24	24	24
ਧ	0.03	0.72	09.0	0.07	0.04
X	8.38	0.97	2.43	15.04	6.35
BATCHES COMPARED	A + C	O +	ы + С	A + D	च + स

EFFECT OF BAKING TREATMENTS UPON SLOW BEND TEST PROPERTIES OF Zn10 PLATED CARBON SPRING STEEL STRIPS. TABLE VIII

CONCLUSIONS	Zn (10) plating caused a highly significant reduction in ductility.	2 hour bake did not significantly improve ductility	18 hour bake did not significantly improve ductility.	After baking for up to 18 hours the	ductility of the placed strips was significantly lower than that of the unplated strips.
SIGNIFICANCE	866<	806>	%06>	>998	<b>%66</b> <
DEGREES OF FREEDOM $(V_1=V_2)$	24	24	24	24	24
Ч	90.0	0.57	0.40	0.08	0.04
W	12.23	66.0	1.06	12.62	89.8
BATCHES COMPARED	A + H	H + I	Н + Л	A + I	A + J

CLASSIFICATION OF SLOW BEND TEST RESULTS FOR Zn2 TABLE IX PLATED CARBON SPRING STEEL STRIPS.

	% of Samples in Embrittled Groups*					
Batch Identification	Not Embrittled	Slight-Moderate Embrittlement	Severe Embrittlement			
A	100	ı				
В	88	12				
С		80	20			
D		80	20			
E	4	88	8			

### \* Classification of Slow Bend Test Data

>109<sup>0</sup> Not embrittled:

Slight-Moderate: 80° - 109° Embrittlement

Severe

Embrittlement: <80°

CLASSIFICATION OF SLOW BEND TEST RESULTS FOR ZnlO PLATED CARBON SPRING STEEL STRIPS. TABLE X

Batch Identification	% of Samples in Embrittled Groups*		
	Not Embrittled	Slight-Moderate Embrittlement	Severe Embrittlement
A	100		
В	88	12	
Н		84	16
I		88	12
J		88	12

## \* Classification of Slow Bend Test Data

>109<sup>0</sup> Not Embrittled:

80° - 109° Slight-Moderate:

Embrittlement

Severe

<80<sup>O</sup> Embrittlement:

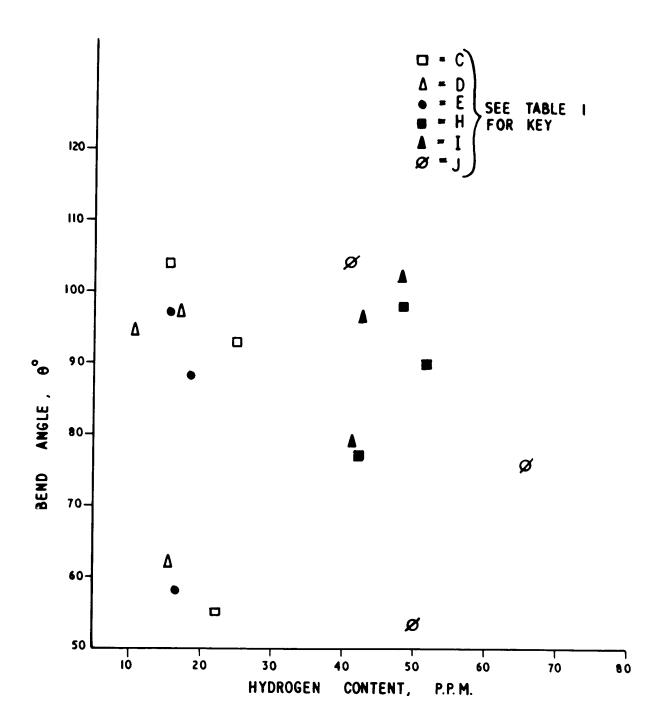
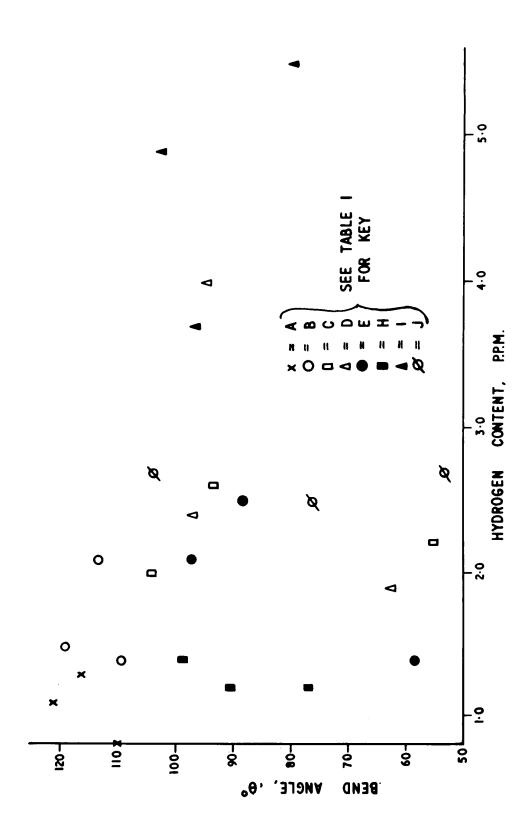


FIG. I. ANGLE AT FRACTURE DURING SLOW BEND TEST

PLOTTED AGAINST HYDROGEN CONTENT OF ZINC

PLATED CARBON SPRING STEEL STRIP.



AGAINST BEND TEST PLOTTED PLATED ZINC BEEN AND UNTREATED, ACID CLEANED FROM WHICH THE ZINC FRACTURE DURING SLOW STRIPS AT CONTENT STEEL

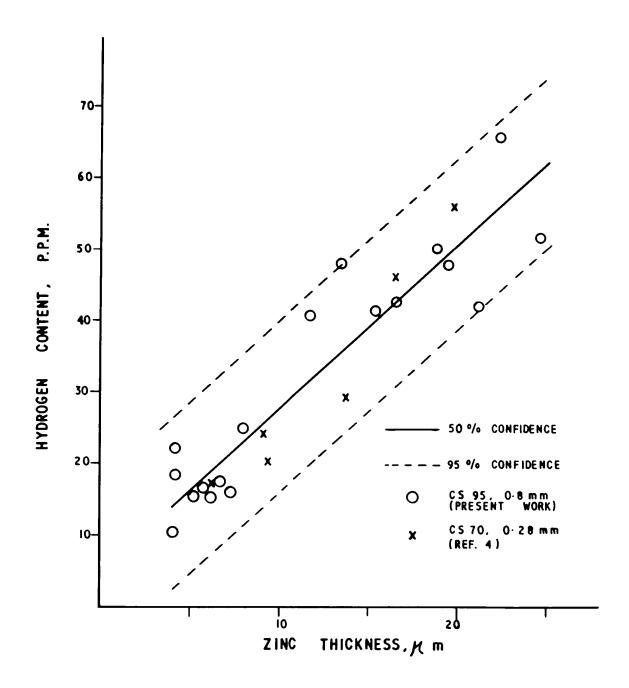


FIG. 3. TOTAL HYDROGEN CONTENT OF ZINC PLATED

CARBON SPRING STEEL STRIP, PLOTTED AS A

FUNCTION OF ZINC THICKNESS.

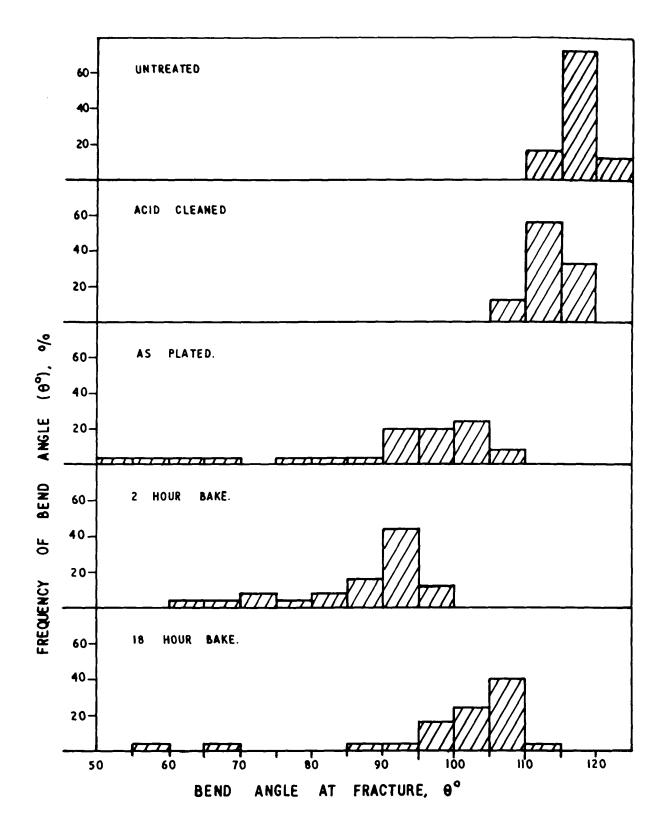


FIG. 4. FREQUENCY DISTRIBUTION OF BEND ANGLES FOR

UNTREATED, ACID CLEANED AND Zn 2 PLATED

CARBON SPRING STEEL STRIPS BEFORE AND AFTER

BAKING AT 190/200° C.

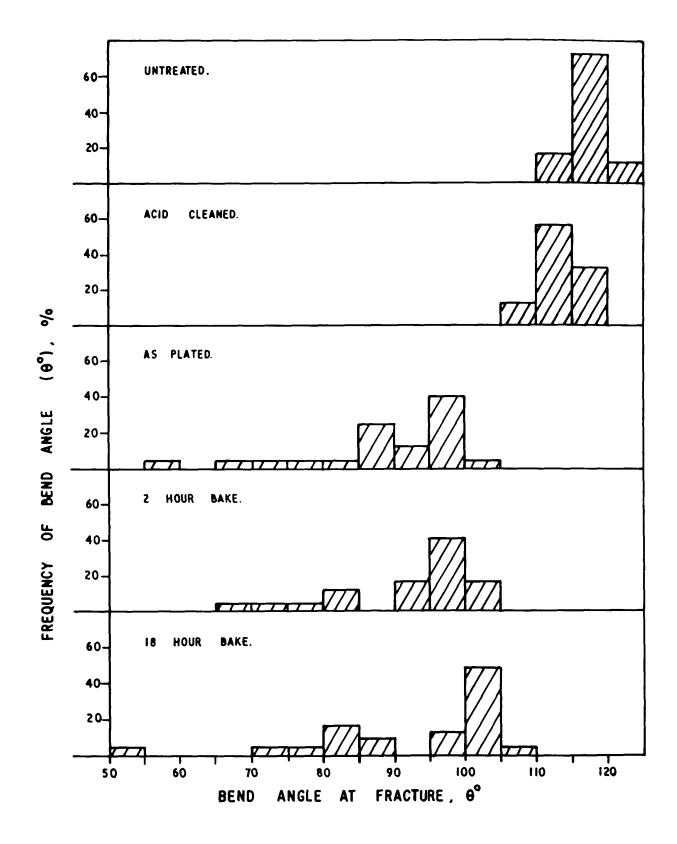


FIG. 5. FREQUENCY DISTRIBUTION OF BEND ANGLES FOR

UNTREATED, ACID CLEANED AND Zn 10 PLATED CARBON

SPRING STEEL STRIPS BEFORE AND AFTER BAKING

AT 190/200°C.

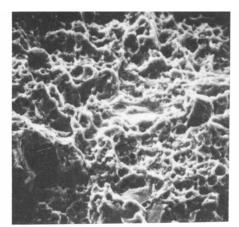


Fig. 6  $\times$  1440 Scanning Electron Fractograph of Unplated Strip showing Ductile Dimpled Fracture. (Sample A5, Bend Angle = 110°).

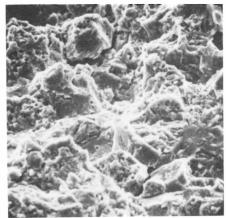


Fig. 7 x 1220

Scanning Electron Fractograph of Zinc Plated Strip showing typical intergranular fracture due to hydrogen embrittlement. (Sample C9, Bend Angle = 55°)