THE SPRING RESEARCH AND MANUFACTURERS' ASSOCIATION

A NOTE OF THE EFFECTS, UPON HYDROGEN EMBRITTLEMENT OF DELAYS BETWEEN ZINC PLATING AND COMMENCEMENT OF DE-EMBRITTLEMENT TREATMENTS

Report No 374

by

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SUMMARY

Samples of CS80 strip at two sulphur contents (0.008%S and 0.047%S) were zinc electroplated, and de-embrittled at 200°C/24 hours after delays of 4, 24, 48 (0.008%S only) and 120 hours between plating and de-embrittlement.

For high sulphur steel, the recovery of ductility by de-embrittlement after a 120 hour delay did not differ significantly from that obtained after a 4 hour delay.

The low sulphur steel was adequately de-embrittled after a 48 hour delay, but there was evidence that the most severely embrittled samples remained brittle if de-embrittlement was delayed for 120 hours after plating.

Commercial treatments of 190°C/18 hours, applied within 30 minutes of plating, gave generally adequate de-embrittlement but the high sulphur steels showed significantly better recovery than the low sulphur steel.

The work suggests that delays of up to 48 hours between plating and de-embrittling may be possible, especially where springs are lightly stressed, but caution should prevail where stress raises, such as holes and stamping flash, are present in electroplated components, especially with regard to low sulphur steels de-embrittled at 190°C.

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1. INTRODUCTION

Several specifications are available relating to the de-embrittlement after zinc plating of high strength spring steel.[1, 2, 3, 4]

Of these specifications, DTD 903D gives explicit instructions that de-embrittlement treatments should commence within 16 hours of zinc plating, whilst the more recent Discussion Document ISO/DIS 2081 (proposed as a revision of BS 1706) suggests that de-embrittlement should commence as soon as possible after plating, and in any event not more than 4 hours after plating is completed.

Recent research at SRAMA into hydrogen embrittlement of hardened and tempered CS80 spring steel has resulted in information concerning the effects upon embrittlement of delays of up to 5 days between the completion of zinc plating and the commencement of a typical de-embrittlement treatment of 200°C/24 hours. The conclusions of this preliminary work form the subject of this report.

2. MATERIALS AND TECHNIQUES

The steels involved in the work were the subject of the most recent SRAMA report, and are identified as Steel 2 (545 Hv) and Steel 3 (515 Hv) containing 0.008% sulphur and 0.047% sulphur respectively.[5]

The composition of the steels are shown in Table I.

The test samples, and techniques used to assess hydrogen embrittlement, have been fully documented in previous SRAMA reports, and consist essentially of a slow bend test of steel strips over a mandrel of specified diameter, with the angle at fracture being taken as a measure of embrittlement.[6]

In the present work test samples of the two steels were slow bend tested in the following conditions:

1. Unplated

2. As zinc plated (by commercial barrel plating via a microprocessor controlled process)

3. Zinc plated and baked, within 30 minutes of plating, at 190°C for 18 hours (a typical commercial treatment)

4. Zinc plated and baked at 200°C for 24 hours after the following delays between plating and de-embrittlement:

   i) 4 hour delay
   ii) 24 hour delay
   iii) 48 hour delay (Steel 2 only)
   iv) 120 hour (5 day) delay

A summary of the test results is presented in Table II.

A considerable amount of statistical analysis, using ANOVA techniques, was involved in this work and it is not proposed to present the data analyses in this report. The results of the analyses can be obtained from SRAMA if required, however.

3. RESULTS AND DISCUSSION

The bend angles of the "as plated" samples of Steel 2 (0.008%S) and Steel 3 (0.047%S) were reduced to 82% and 41% of the unplated values respectively, thus confirming that embrittlement had occurred, and that the low sulphur Steel 2 was less severely embrittled than the high sulphur Steel 3 material.

For each steel de-embrittled at 200°C for 24 hours, analysis of variance showed that there was no significant difference in the bend test results for delays, between plating and de-embrittlement, of up to 120 hours.

There was evidence that, for 4 hours and 24 hours delay, the higher sulphur steel showed the best response to de-embrittlement. Although the two steels apparently responded equally after delays of 120 hours, there was evidence that some embrittlement remained in the more severely embrittled samples of the low sulphur steel. In general, however, the response of the steels to de-embrittlement, at 200°C for 24 hours, did not vary greatly with sulphur content, thus confirming the most recent work at SRAMA.[5]

The high sulphur steel showed no significant difference in response to de-embrittlement at 190°C for 18 hours and 200°C for 24 hours. The low sulphur steel, however, responded better to the 200°C treatment than to the 190°C treatment.

The differing response of the two steels to de-embrittlement was particularly notable when they were compared after treatment at 190°C for 18 hours. In this case, the improvements in ductility of the high sulphur steel were noticeably higher than those for the low sulphur steel, the difference in behaviour being significant at the 99% level of the 'F' distribution.

Whilst this work suggests that, especially for high sulphur steels, delays of up to 48 hours may be tolerated between plating and the commencement of de-embrittlement at 200°C for 24 hours, it should be emphasized that the results relate to samples which were free from the holes, stamping flash and other stress raisers which will almost inevitably be present in plated spring components. In consequence, extension of the present laboratory results to industrial spring components will require that verification trials be undertaken to establish whether or not stress raisers will impair the ductility of the components if delays of greater than 4 hours, between plating and de-embrittlement, are anticipated. Such verification will be particularly important for springs made from low sulphur steels.

This research is continuing under a program of work between SRAMA and Sheffield University financed jointly by SRAMA members, the Department of Industry and the Science and Engineering Research Council.

4. CONCLUSIONS

1. Delays of up to 120 hours, between zinc plating and commencement of de-embrittlement at 200°C/24 hours, appeared to have no significant effect upon restoration of ductility for high sulphur hardened and tempered carbon spring steel (0.047% S).

2. The ductility of similarly de-embrittled low sulphur spring steels (0.008%S) was adequately restored after delays of up to 48 hours between plating and de-embrittling. Delays of up to 120 hours were less desirable for the low sulphur steel, however, there being some evidence that such delays were associated with less effective recovery of ductility for the more severely embrittled samples.

3. High sulphur steel (0.047%S) was more effectively de-embrittled than low sulphur steel (0.008%S), this difference being more marked for commercial de-embrittlement at 190°C/18 hours.

4. In general terms, delays of up to 48 hours, between plating and de-embrittling may be possible for slightly stressed components, but caution should be exercised where stresses are high or stress raisers are present.

   Particular emphasis should be placed on such caution for low sulphur steels de-embrittled at 190°C.

5. <u>REFERENCES</u>

1. BS 1706: 1960 "Specification for Electroplated Coatings of Cadmium and Zinc on Iron and Steel".

2. DTD 903D: 1966 "Process Specification: Zinc Plating".

3. DEF 03-4/Issue 2: 1977 "The Pre-treatment and Protection of Steel Parts of Specified Maximum Tensile Strength Exceeding 1450 N/mm^2".

4. ISO/DIS 2081: 1982 "Draft British Standard Methods of Specifying Electroplated Coatings of Zinc on Iron and Steel (Revision of BS 1706)".

5. Reynolds, L F, " Hydrogen Embrittlement of Hardened and Tempered Carbon Spring Steel Strip Electroplated with Zinc. Fourth Progress Report: The Effect of Steel Composition on the Embrittlement and De-embrittlement of CS80 Steel". SRAMA Report 365, March 1984.

6. Reynolds, L F, ibid, "First Progress Report: The Effects of Hydrogen Content, Zinc Thickness and Baking Time upon Hydrogen Embrittlement". SRAMA Report 345, March 1982.

TABLE I CHEMICAL COMPOSITION OF CARBON SPRING STEEL STRIP

Steel Identification	Element, %								
	C	Si	Mn	P	S	Cr	Mo	Ni	Cu
2	0.77	0.31	0.67	0.011	0.008	0.13	0.02	0.06	0.07
3	0.81	0.28	0.62	0.008	0.047	0.06	0.02	0.13	0.16

TABLE II BEND ANGLE RESULTS (DEGREES) FOR CS80 STEEL 2 (0.008%S) AND STEEL 3 (0.047%S)

Mandrel: 4.53 mm diameter

Strip Condition	Steel 2			Steel 3		
	Mean	Standard Deviation	Number of Samples	Mean	Standard Deviation	Number of Samples
Unplated	85.0	1.7	20	99.2	3.8	25
"As plated" tested 4 hours after plating	70.1	6.6	15	40.7	7.6	15
"As plated" tested 24 hours after plating	69.9	3.6	15	40.3	5.5	15
De-embrittled 190°C/18 hours within 30 mins of plating	75.8	3.3	12	96.3	5.1	12
De-embrittled 200°C/24 hours after 4 hour delay	80.1	1.4	15	96.1	4.6	15
Ditto, after 24 hour delay	80.3	2.7	17	97.5	7.8	16
Ditto, after 48 hour delay	79.4	2.8	20	-	-	-
Ditto, after 120 hour delay	78.4	5.5	16	94.3	3.8	16