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# HYDROGEN EMBRITTLEMENT OF HIGH STRENGTH STEEL ELECTROPLATED WITH ZINC-COBALT ALLOYS

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**Abstract;** Slow strain rate tests were performed on quenched and tempered AISI 4340 steel to measure the extent of hydrogen embrittlement caused by electroplating with zinc-cobalt alloys. The effects of bath composition and pH were studied and compared with results for electrodeposited cadmium and zinc-10%nickel. It was found that Zinc-1%cobalt alloy coatings caused serious hydrogen embrittlement (EI 0.63); almost as severe as that of cadmium (EI 0.78). Baking cadmium plated steel for 24 hours at 200°C gave full recovery of mechanical properties but specimens plated with zinc-1%cobalt and then baked still failed in 89% of the time of unplated controls. It was shown that hydrogen uptake and embrittlement could be controlled by depositing thin layers of cobalt or nickel at the steel/coating interface. For example, the least embrittlement was caused by zinc-10%nickel (EI 0.037) due to a nickel rich layer with very low hydrogen diffusion coefficient that formed during the initial stages of electroplating. Similarly, a 0.5 micron nickel layer was effective in lowering the embrittlement caused by zinc-1%cobalt to that of zinc-10%nickel. Furthermore, a 0.5 micron cobalt layer deposited before a zinc-1%cobalt coating gave virtually 100% recovery of mechanical properties after baking.

## INTRODUCTION

Electrodeposited zinc-cobalt alloys are possible alternatives to cadmium for sacrificial corrosion protection of steel components. The coatings contain typically 1-4% Co by weight and are produced by an anomalous co-deposition mechanism [1,2] that promotes a zinc-rich alloy by suppressing the reduction of the more noble cobalt. The corrosion rate of electrodeposited zinc-4%Co is reported to be very similar to that of the more expensive zinc-14%Ni [3].

The possibility of hydrogen embrittlement is an important consideration when high strength steels are electroplated. Plating processes generally have a current efficiency of less than 100% and a proportion of the current passed during electrodeposition results in the generation of hydrogen, some of which becomes incorporated in the deposit and subsequently diffuses into the substrate. When hydrogen embrittlement is thought to be a risk a de-embrittlement treatment may be required. Following cadmium plating, for example, steels with a tensile strength over 1800 MPa are baked for 24 hours at a temperature in the range 190-230°C [4].

The aim of the work described in this paper was to investigate the extent to which electroplating with zinc-cobalt alloys causes hydrogen embrittlement of high strength steel. Slow strain rate tests were carried out on plated tensile specimens to measure the effect of the absorbed hydrogen on the loss of the steel's mechanical properties and the effectiveness of post-plating baking treatments in restoring these properties. For comparison, further tests were conducted on specimens plated with pure zinc, Zn-10%Ni and cadmium and also with Zn-12%Co-9%Fe to investigate ways in which hydrogen uptake could be controlled by the coating composition.

## METHODS

### Materials

The high strength steel AISI 4340 was selected for the slow strain rate tests due to its known susceptibility to hydrogen embrittlement in the quenched and tempered condition. The composition of the material is shown in Table 1.

**Table 1 Composition of AISI 4340 Steel**

| %C | %Mn | %Si | %Cr | %Ni | %Mo | %Al | %Cu | %S | %P |
|---|---|---|---|---|---|---|---|---|---|
| 0.41 | 0.79 | 0.23 | 0.88 | 1.67 | 0.22 | 0.024 | 0.13 | 0.003 | 0.009 |

### Zinc-Cobalt Electroplating

Zinc-cobalt electrodeposits were produced from an acid sulphate bath of composition [2] shown in Table 2. The electrolyte contained 8% Co and was similar to that used commercially, except that no brighteners were added. A small quantity of the surfactant Teepol was included to avoid the formation of hydrogen bubbles that could lead to defects in the coating.

**Table 2 Composition of the zinc-cobalt plating bath**

| Addition | Concentration (mol $dm^{-3}$) |
|---|---|
| $ZnSO_4$ $7H_2O$ | 1.739 |
| $CoSO_4$ $7H_20$ | 0.17 |
| $NaSO_4$ | 0.352 |
| $CH_3COONa$ | 0.11 |
| Teepol | 0.001 |

The standard bath condition, which was used for the majority of the experiments, was a pH of 4.2 and a temperature of 50°C. The resulting coatings were even and shiny, with a maximum cobalt content of 1%. Plating for 15 minutes at a current density of 10 $mAcm^{-2}$ gave an average coating thickness of 8 μm, which is generally specified for threaded components.

The cadmium plating was carried out by a commercial electroplater and the coatings of pure zinc, zinc-10%nickel and zinc-12%cobalt-9%iron were plated in the laboratory from the electrolytes shown in Table 3.

**Table 3 Compositions of the other plating solutions**

| Coating | Current | Time | pH | Temp | Electrolyte | Concentration |
|---|---|---|---|---|---|---|
| | ($mAcm^{-2}$) | (mins) | | (°C) | | (mol $dm^{-3}$) |
| 100% Zn | 10 | 15 | 1 | 50 | $ZnSO_4.7H_2O$<br>$H_2SO_4$ | 1.2<br>0.6 |
| 92-89% Zn<br>8-11% Ni | 20 | 15 | 5 | 25 | $ZnSO_4.7H_2O$<br>$NiSO_4.7H_2O$ | 0.92<br>0.08 |
| 79% Zn<br>12% Co<br>9% Fe | 30 | 10 | 3 | 43 | $ZnSO_4.7H_2O$<br>$CoSO_4.7H_2O$<br>$FeSO_4.7H_2O$<br>$Na_2SO_4.7H_2O$<br>$CH_3COONa.3H_2O$<br>$H_2SO_4$ | 0.4<br>0.4<br>0.06<br>2.5<br>0.3<br>0.5 |

**Hydrogen Embrittlement Tests**

Tensile specimens for slow strain rate embrittlement testing were machined by CNC turning from normalised 6mm diameter rod to give a 25mm gauge length of 2.25mm diameter. They were then hardened by heating in a neutral salt bath at 835–840°C for 15 minutes, followed by an oil quench at room temperature. Tempering was carried out at 250°C for 2 hours to give a mean hardness of 528 $HV_{10}$ and a mean tensile strength of 1850 $MNm^{-2}$. The tempering temperature of 250°C was chosen to avoid further change in mechanical properties when the specimens were later de-embrittled by baking for 24 hours at 200°C.

The specimens were prepared for plating by abrading with silicon carbide paper, followed by degreasing in isopropanol. Pickling and anodic or caustic cleaning were not used to avoid hydrogen uptake. The slow strain rate tests were started immediately after plating, except for those specimens commercially plated with cadmium, which were stored in liquid nitrogen to prevent loss of absorbed hydrogen until they could be tested.

The specimens were tested on a Cortest slow strain rate machine at a strain rate of $1.1 \times 10^{-6}$ $s^{-1}$ and the times to failure were recorded. In all, twenty-five specimens were tested in the unplated condition to act as controls. The extent of the embrittlement caused by each plating treatment was assessed by carrying out slow strain rate tests on five or more specimens in the as-plated condition. Additionally, the effectiveness of thermal de-embrittlement was measured by testing further specimens after first plating and then baking for 24 hours at 200°C.

## RESULTS

Hydrogen embrittlement tests often display a range of times to failure when carried out under apparently identical experimental conditions. This is due to the variation in the number, size and distribution of microstructural flaws within the material at which hydrogen accumulates and leads to fracture. In this work Weibull statistics was used to distinguish between the effects of each of the experimental variables.

**Weibull Model of Failure Times**

It can be shown that for a Weibull model the probability, $P_s$, of a specimen not failing within time, t, is given by;-

$$P_s = 1 - P_t = e^{-xt} \qquad [1]$$

Where $P_t$ is the probability of failure and x is a shape parameter termed the Weibull slope [5-7]. In the present context x represents the probability per unit time that during time, t, a crack will form in the specimen of sufficient size to cause failure. The value of x can be obtained from the negative gradient of a graph of ln $P_s$ against t. A schematic Weibull plot of failure times is shown in Fig 1. The graph is displaced along the time axis by the amount $t_i$, known as the minimum crack incubation time. (When $P_s = 1$, then ln $P_s = 0$ and $t = t_i$ ).

Equation [3] can be modified to include $t_i$ .

$$P_s = 1 - P_t = e^{-x(t - ti)} \qquad [2]$$

**Unplated Specimens**

A Weibull plot of the times to failure in the slow strain rate tests for specimens that had been quenched and tempered but not electroplated is shown in Fig 2. All of the specimens failed in times ranging between 17 and 21 hours with a mean time of 18.9 hours.

**Effect of Zinc-Cobalt and Cadmium Plating**

The effect of electroplating specimens with 8 microns of cadmium or zinc-1%cobalt was to promote hydrogen embrittlement of the steel and reduce the times to failure. The results for these two coatings are compared in Fig 3. The cadmium caused more embrittlement with a mean time to failure of 4.2 hours compared to 7.0 hours for the zinc-cobalt.

After baking at 200°C for 24 hours to promote loss of hydrogen from the surface, specimens that had been plated with cadmium showed full recovery of mechanical properties and the mean time to failure was restored to that of the unplated controls (Fig 4). The statistical Student t-test [8] was used to confirm (95% confidence) that the failure times of the baked cadmium plated specimens and the control specimens were part of the same population. Similarly, it was demonstrated that the baking heat-treatment did not affect the times to failure of the control specimens. In contrast, baking the zinc-cobalt plated specimens for 24 hours gave incomplete recovery and the times to failure were still only 89% of those for the controls. Additional tests were performed in which the baking time was increased to 48 hours but their recovery only increased to 91%.

The pH of the zinc-cobalt plating bath had an influence on the times to failure, as shown by the results in Table 4. Raising the pH from the standard condition of 4.2 to a value of 5 decreased the time to failure whereas plating at pH 1, which might have been expected to increase the hydrogen uptake, had the effect of reducing the hydrogen embrittlement. The reasons for these changes are discussed later.

**Table 4 Effect of Zn-Co bath pH on failure times in slow strain rate tests**

| Bath pH | Time to Failure (hours) |
|---|---|
| 1 | 7.2 |
| 3 | 6.3 |
| 4.2 | 7.0 |
| 5 | 4.5 |

**Comparison with Pure Zinc Plating**

In order to investigate the embrittlement promoted by zinc-cobalt coatings in more detail, comparison was made with the times to failure of specimens electroplated with pure zinc. It was found that pure zinc promoted more embrittlement than zinc-1%cobalt, with a mean time to failure of 5.6 hours, as shown in Fig 5. The presence of just 1%Co in the zinc-cobalt alloy was beneficial in reducing the extent of embrittlement. Like the zinc-1%cobalt coating, baking the pure zinc plated specimens did not fully restore their mechanical properties and they failed in 87% of the time of the unplated controls.

**Zinc-10%Nickel Plating**

The results for electroplated Zn-10%nickel are included in Fig 5. The mean time to failure of 18.2 hours indicates that very little hydrogen embrittlement was promoted by this coating. It had been shown in an earlier study [9] that the reason that zinc-nickel alloys cause only a low level of embrittlement is that the first material to deposit on the surface of the steel has a higher nickel content than the remainder of the coating. Nickel has a very low diffusion coefficient for hydrogen [10], in the order of $8 \times 10^{-10}$ $cm^2s^{-1}$, and the nickel-rich interlayer acts as a barrier to the diffusion of hydrogen from the coating into the steel.

In the case of the zinc-1%cobalt coatings it was expected that a cobalt-rich layer would form at the coating/substrate interface. If cobalt also acts as a barrier to hydrogen then this would explain why these coatings caused less hydrogen embrittlement than pure zinc coatings.

**Effects of Nickel and Cobalt Interlayers**

To test this hypothesis tests were carried out in which the slow strain rate specimens were electroplated by a double bath treatment (DBT); first with a 0.5 micron interlayer of either pure cobalt or pure nickel, followed by 8 microns of zinc, zinc-10%cobalt or zinc-10%nickel. The results for each of these coatings are shown in Fig 6. The least embrittlement was caused by electroplating a zinc-10%nickel coating over a 0.5 micron layer of nickel, confirming the beneficial effects of nickel as a barrier to hydrogen diffusion. A particularly significant result is that the nickel interlayer was also effective in lowering the embrittlement of a zinc-cobalt coating to that of zinc-nickel. This could have important practical benefits as a means of producing zinc-cobalt coatings with the minimum risk of causing hydrogen embrittlement.

The cobalt interlayer also appeared to reduce the extent of embrittlement but it was not nearly as effective as the interlayer of nickel (Fig 6). The results for zinc-cobalt deposited over 0.5 microns of cobalt were very similar to those for zinc over cobalt and to those of zinc-cobalt alone. Surprisingly zinc-nickel, when plated by itself, caused little embrittlement but when coated over cobalt gave a similar amount of embrittlement to these other coatings. It would appear that in each case the level of embrittlement was controlled by the hydrogen trapped in the cobalt layer at the steel surface rather than that in the remainder of the coating.

**Influence of Baking Treatment**

The effects of baking the zinc-cobalt coatings with the cobalt and nickel interlayers are shown in Fig 7. An important observation was that in both cases virtually full recovery was achieved. As baking the zinc-cobalt coating that did not have an interlayer failed to restore its properties to those of the unplated controls it is assumed that the presence of a thin layer of cobalt can have a significant effect. It is thought that a cobalt interlayer acts as a sufficient barrier to hydrogen that during baking the hydrogen in the coating escapes to atmosphere rather than diffusing into the steel.

**Zinc-Cobalt-Iron Plating**

The results for the zinc-12%cobalt-9%iron alloy were very promising as shown in Fig 8. This coating was the least embrittling of the zinc-cobalt alloys tested with a mean time to failure of 10.8 hours compared to 5.6 hours for zinc, 7.0 hours for zinc-1%cobalt and 7.2 hours for zinc-cobalt plated over a cobalt interlayer.

**Embrittlement Indices**

The extent of hydrogen embrittlement caused by a particular process is frequently assessed by comparing values of the embrittlement index (EI), which is defined as;-

$$EI = 1 - (t_p/t_c) \qquad [3]$$

where $t_p$ and $t_c$ are the times to failure of the plated specimens and controls respectively. The mean embrittlement indices for the different plating processes are compared in Table 5. Clearly, the indices depend on the susceptibility of the steel in addition to the tendency of the electrodeposit to promote hydrogen uptake but values above 0.6 are generally taken to indicate severe embrittlement.

**Table 5 Embrittlement indices for each plating process**

| Coating | Embrittlement Index |
|---|---|
| Cadmium | 0.78 |
| Zn | 0.7 |
| Zn-1%Co (pH 4.2) | 0.63 |
| Zn-1%Co baked | 0.11 |
| Zn-Co-Fe | 0.43 |
| Zn-10%Ni | 0.037 |

## DISCUSSION

The results show that electrodeposited Zn-1%Co coatings promote high levels of hydrogen embrittlement in a susceptible high strength steel substrate (Fig 3). This embrittlement was much greater than that caused by Zn-10%Ni plating but not quite as severe as that for pure zinc or cadmium (Figs 3,5 & Table 5).

**Noble Metal Enrichment**

The lower risk of embrittlement from zinc-10%nickel coatings is attributed to the deposition of a nickel-rich layer in the first stages of electroplating [9]. Nickel is an effective barrier to hydrogen uptake by the steel from the coating as it has a very low coefficient for hydrogen diffusion [10]. This conclusion is supported by the results for the dual bath treatments in which a 0.5 micron layer of nickel deposited beneath a zinc-1%cobalt layer reduced hydrogen embrittlement to the level caused by zinc-10%nickel alone (Fig 6).

The nickel-rich layer forms initially during electrodeposition by a 'normal' plating process as nickel is electrochemically a more noble element than zinc. It is only after the pH at the cathode surface has risen to 5.3 that whiskers of zinc hydroxide form, suppressing nickel deposition and allowing zinc to be reduced and deposited by the anomalous codeposition mechanism [11].

It was expected that a cobalt-rich layer would form in a similar way in the early stages of zinc-cobalt electroplating and that the presence of this layer was responsible for the partial reduction in the severe embrittlement caused by pure zinc coatings. Indeed, a cobalt-rich layer, containing 7%Co, was detected at the coating/substrate interface using EDX and Auger electron spectroscopy [12] and other published work on a similar electroplated zinc-cobalt alloy reported that an initial layer containing 10% cobalt forms in the early stages of plating [13]. In the present study, thicker cobalt-rich layers were formed in deposits plated at a lower bath pH, due to the longer time needed to establish the surface pH of 5.3 for the onset of anomalous codeposition. The longest time to failure and the lowest hydrogen uptake occurred with deposits plated at pH 1, where the thickest cobalt-rich layer was formed. Conversely, the shortest times and highest hydrogen uptake corresponded to plating at pH 5 at which the least cobalt enrichment took place.

**Effectiveness of Baking Treatment**

The results in Fig 4 showed that although the baking treatment was effective in removing absorbed hydrogen and reducing the risk of embrittlement in cadmium plated steel it did not give full recovery with zinc-cobalt alloy coatings. The specimens still failed in 89% of the time of the unplated controls, which was similar to the behaviour of specimens plated with pure zinc. Even after baking, the risk of an embrittlement failure in a zinc-cobalt plated specimen was greater than that for zinc-10%Ni plating without a baking treatment (Table 5).

It is possible that the zinc and zinc-cobalt electrodeposits were in a form that was relatively impervious to hydrogen so that it did not all escape to atmosphere during baking. In these circumstances, a proportion of the hydrogen would remain trapped in the steel and may lead to a delayed failure when a stress is subsequently applied.

The composition of the first deposit to form on the substrate has a controlling effect both on hydrogen uptake and its loss from the surface during baking. When a 0.5micron cobalt interlayer was deposited beneath a zinc-1%cobalt coating nearly 100% recovery of properties was achieved by baking. The cobalt interlayer is thought to have acted as a barrier to hydrogen so that during baking the hydrogen in the coating escaped to atmosphere rather than diffusing into the steel.

## CONCLUSIONS

[1] Zinc-1%cobalt alloy coatings electrodeposited on a quenched and tempered AISI 4340 high strength steel substrate resulted in serious hydrogen embrittlement of the steel (Embrittlement Index 0.63). However, this embrittlement was less severe than that caused by cadmium or zinc electroplating (Embrittlement Indices 0.78 and 0.7 respectively).

[2 ] The least embrittlement from zinc-1%cobalt occurred when the coatings were deposited at pH1 due to the formation of a cobalt-rich layer at the coating/substrate interface. In contrast, the greatest embrittlement arose from plating at pH 5 when the cobalt-rich layer was not detected.

[3] The mechanical properties of cadmium electroplated specimens recovered fully after a 24 hour baking treatment at 200°C, whereas specimens that had been plated with zinc-1%cobalt and then baked still failed in 89% of the time of unplated controls.

[4] Even after a baking treatment, the risk of an embrittlement failure in a zinc-1%cobalt plated specimen was greater than that caused by an unbaked zinc-10%nickel plating.

[5] A 0.5 micron cobalt interlayer deposited before the zinc-1%cobalt coating gave virtually 100% recovery of mechanical properties after baking. It is thought that the cobalt layer acted as a sufficient barrier to hydrogen that during the baking treatment the hydrogen in the coating escaped to atmosphere rather than diffusing into the steel.

[6] Of the coatings tested, the least hydrogen embrittlement was caused by electroplated zinc-10%nickel (Embrittlement Index 0.037). This was attributed to the formation of a layer at the coating/substrate interface that was enriched in nickel, which is known to have a very low hydrogen diffusion coefficient.

[7 ] A 0.5 micron nickel interlayer was effective in lowering the embrittlement caused by zinc-1%cobalt to that of zinc-10%nickel. This finding offers a practical method of reducing the risk of embrittlement associated with zinc-cobalt coatings.

[8] The results for the zinc-12%cobalt-9%iron alloy were very promising as this coating caused the least embrittlement of the zinc-cobalt alloys tested (Embrittlement Index 0.43).

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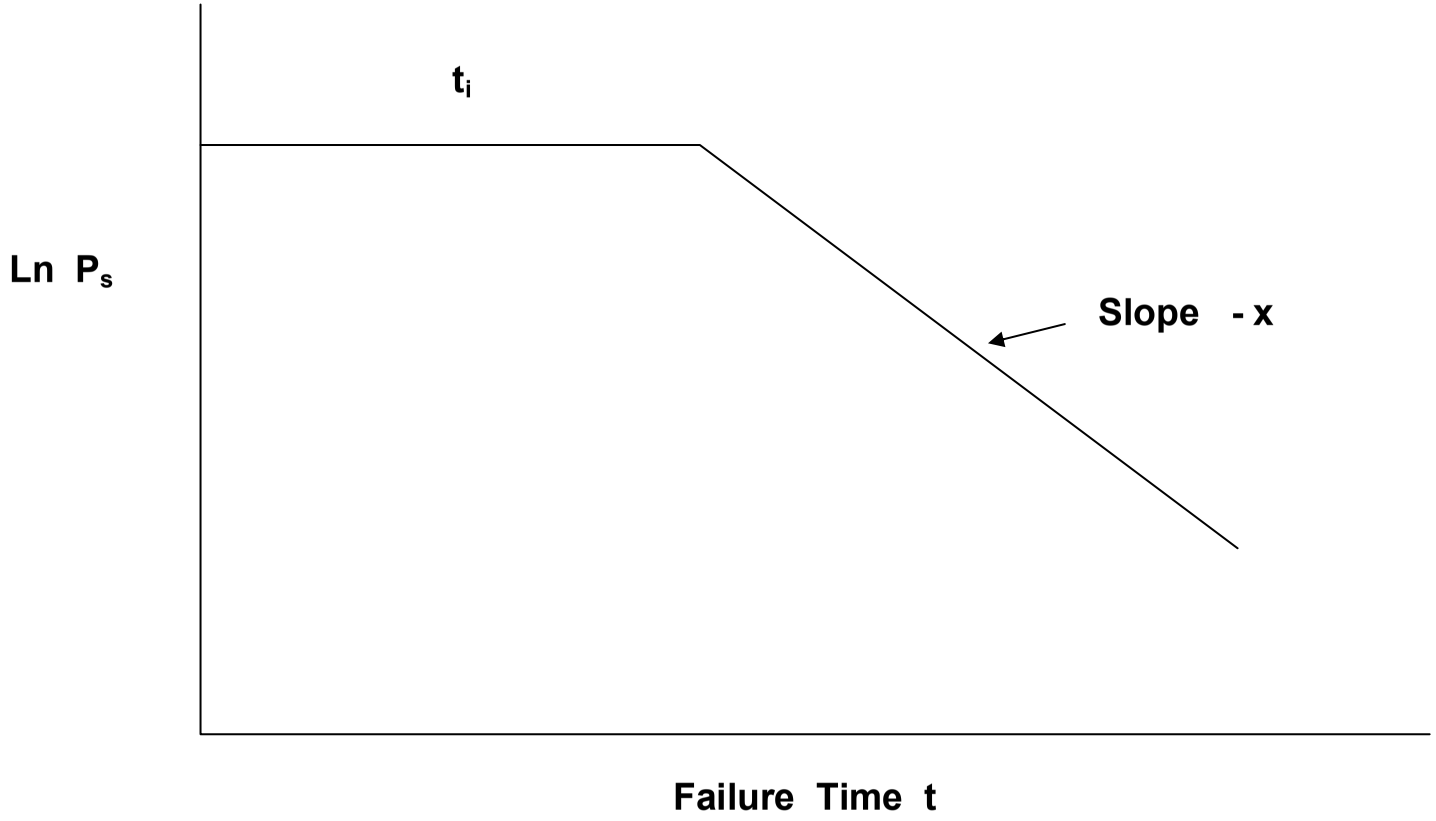
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**Fig 1 Schematic Weibull plot showing the probability of survival, $P_s$, against failure time, t**




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**Fig 2 Weibull plot of failure times in the slow strain rate test for unplated control specimens**

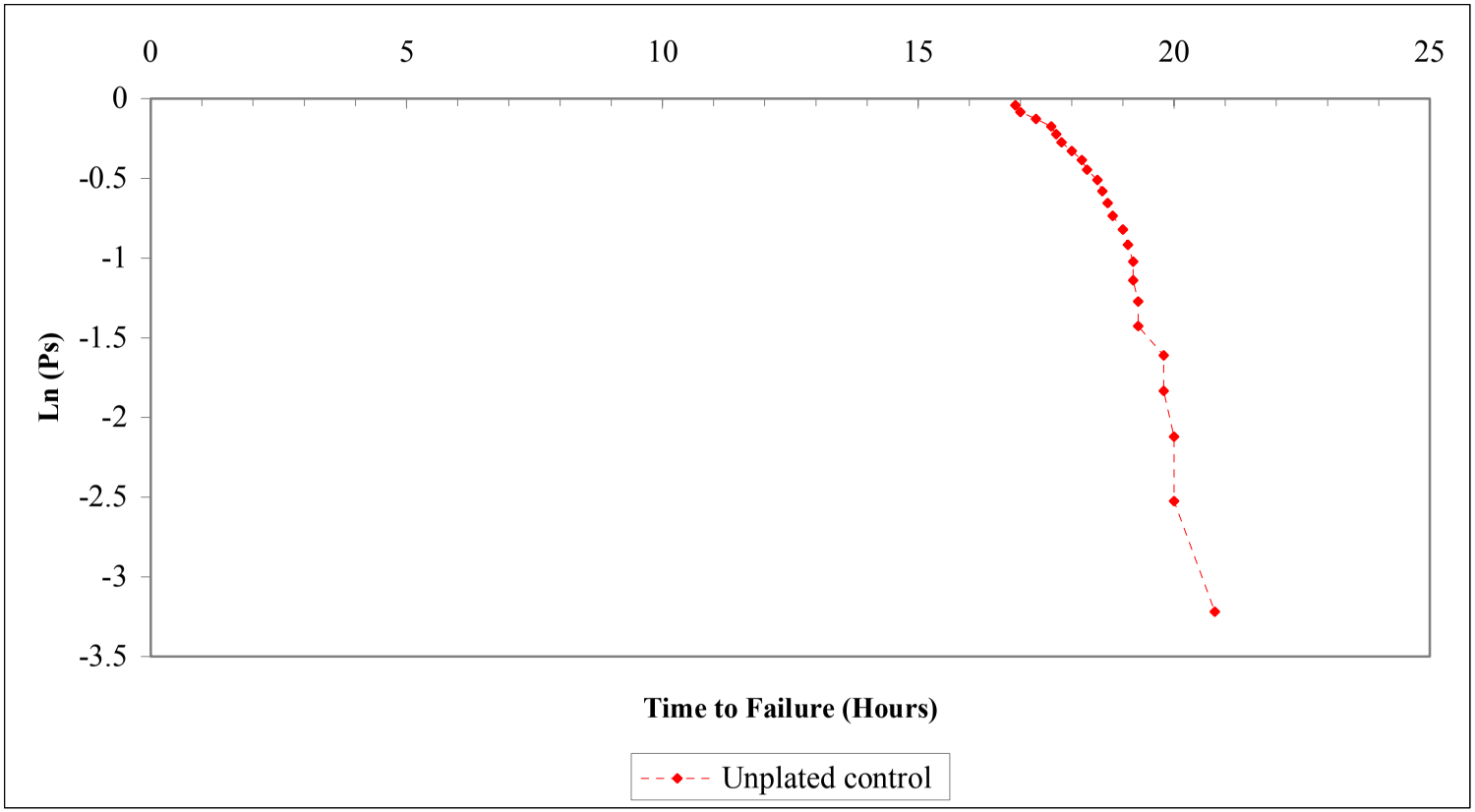


**Fig 3 Weibull plot showing the effect of cadmium and zinc-1%cobalt plating on the time to failure**

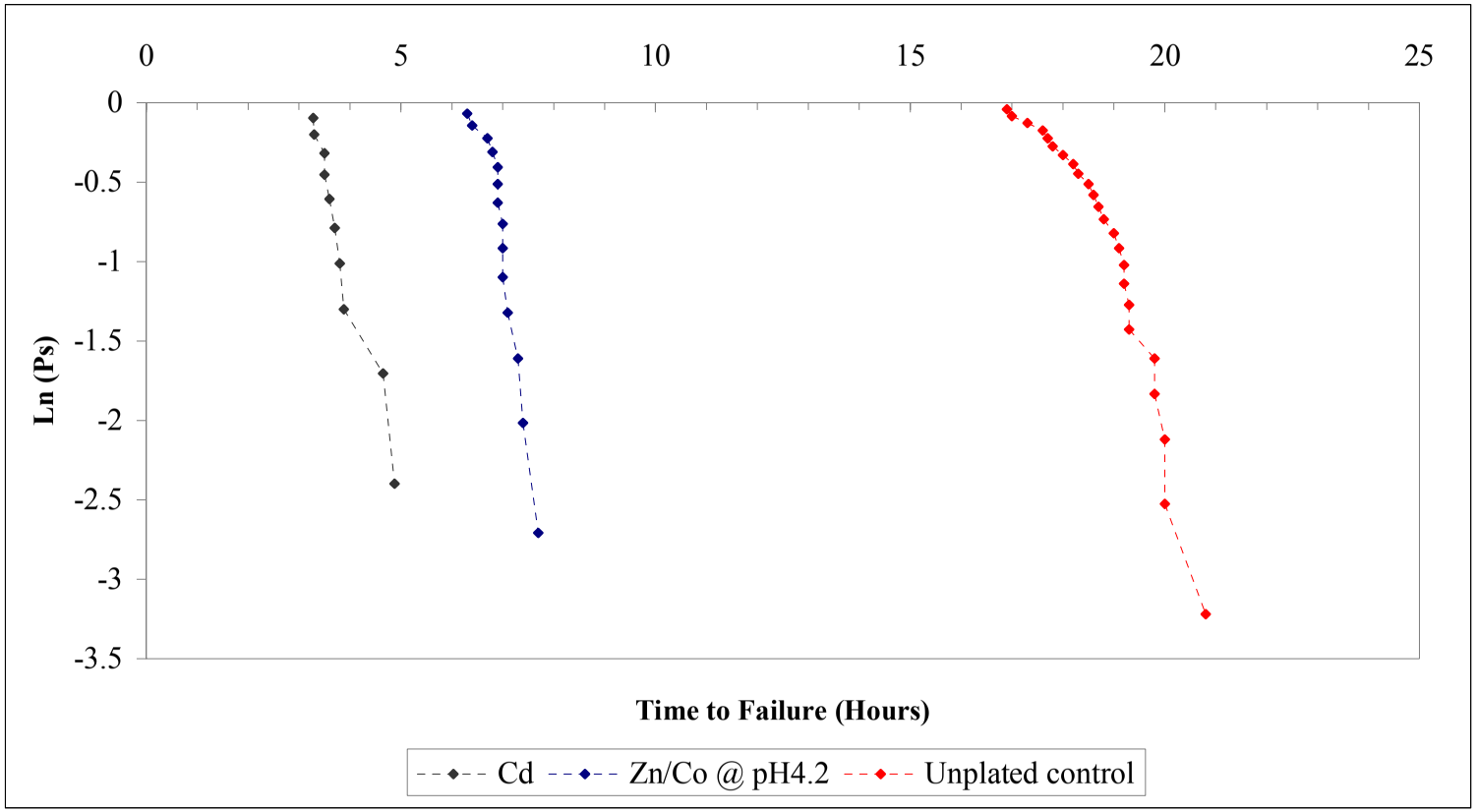


**Fig 4 Weibull plot showing the effect of baking on the failure times of cadmium and zinc-1%cobalt plated specimens**

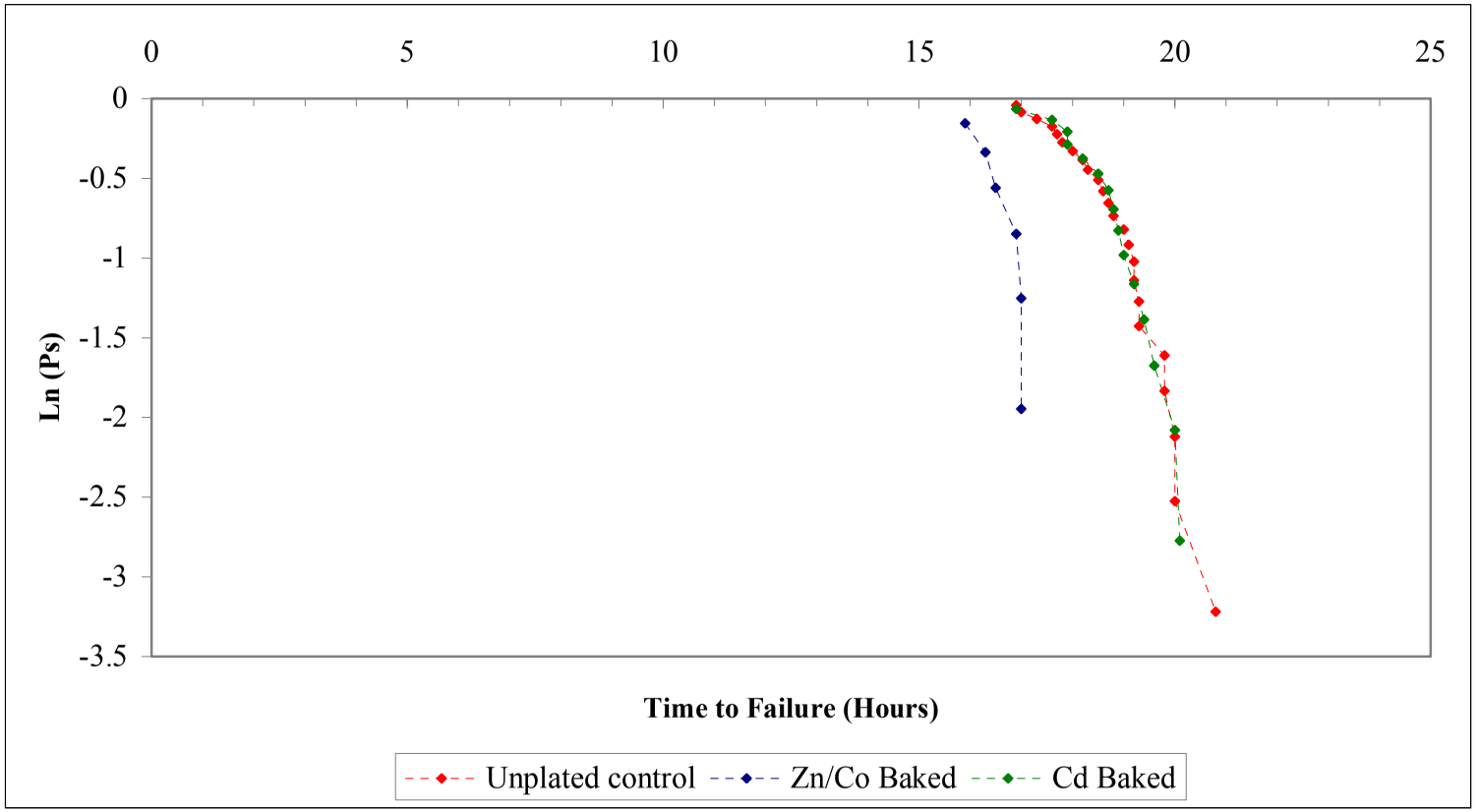


**Fig 5 Weibull plot comparing the failure times of specimens plated with Zn-1%Co, zinc and Zn-10%Ni**

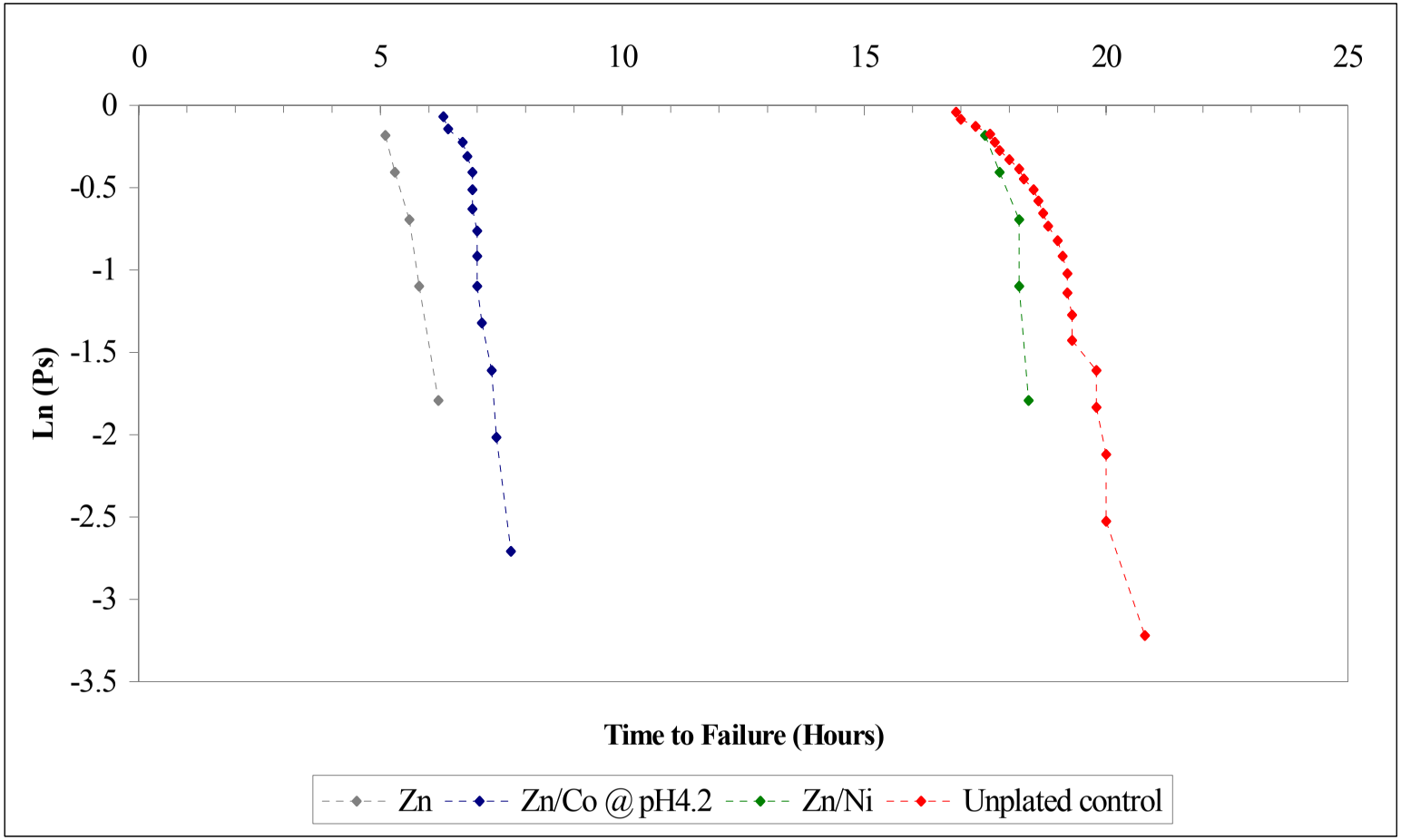


**Fig 6 Weibull plot showing the effect of an interlayer of nickel or cobalt on the times to failure**

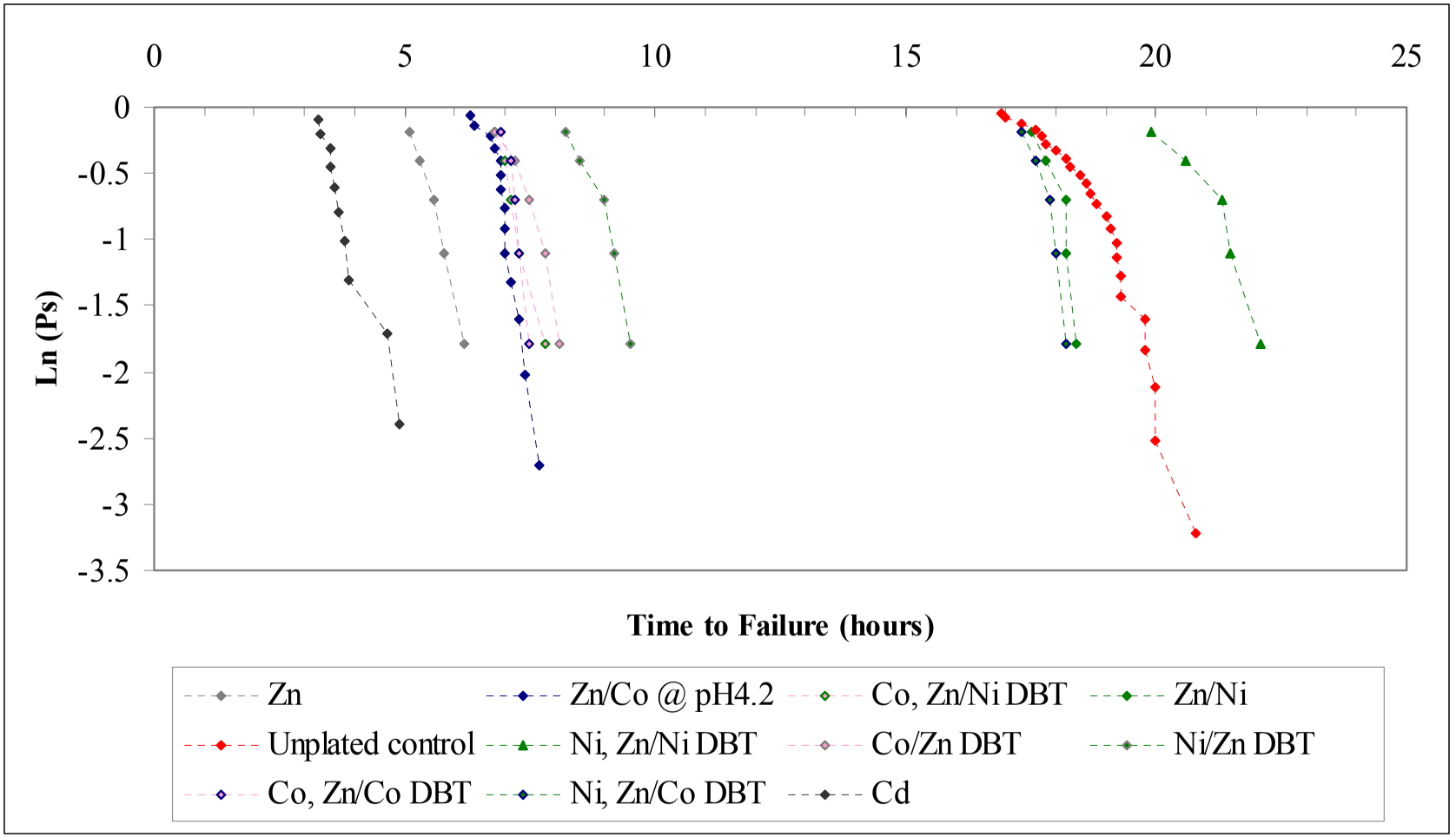


**Fig 7 Weibull plot showing the effects of baking on the times to failure of zinc-cobalt coatings with nickel or cobalt interlayers**

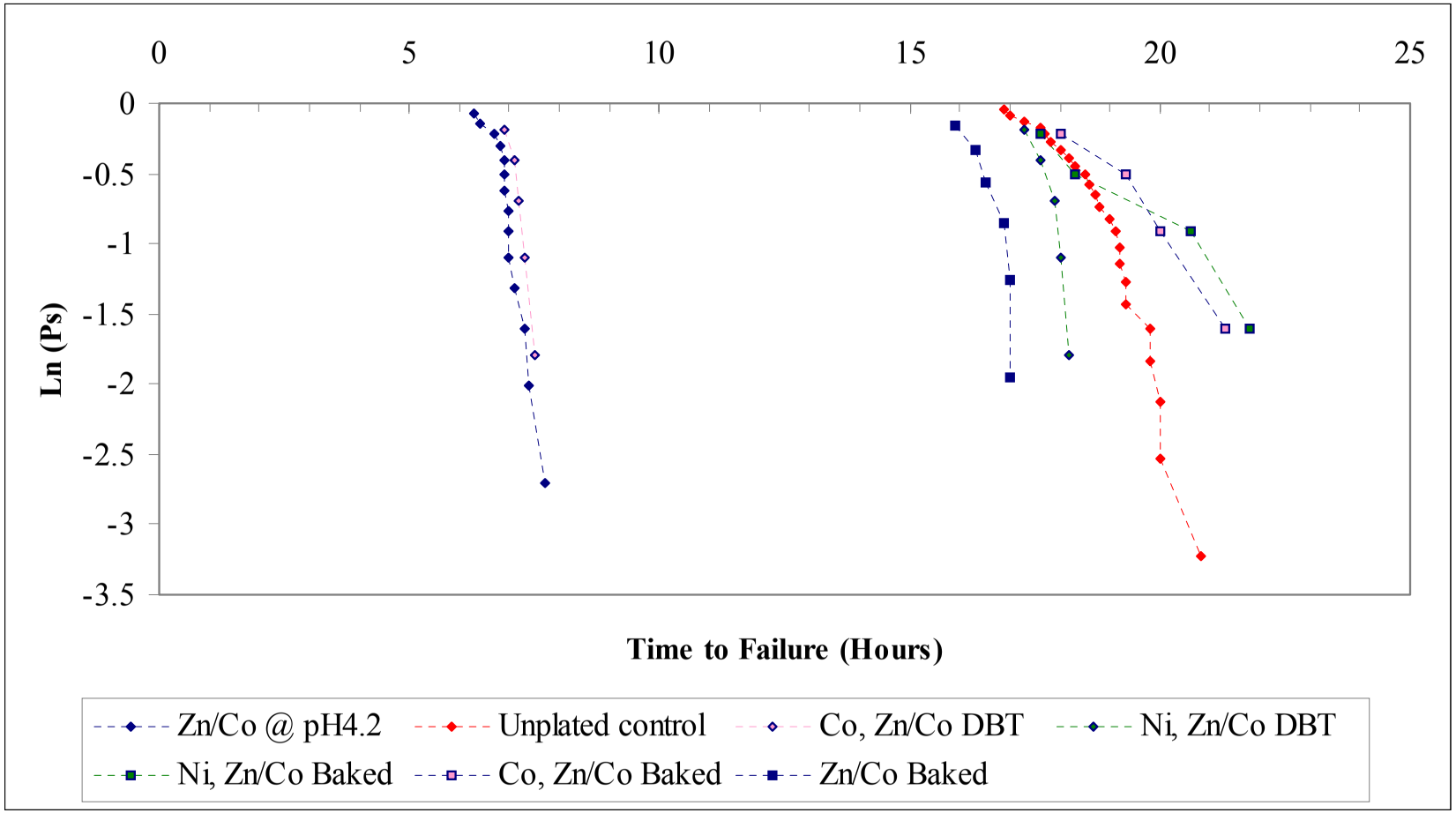
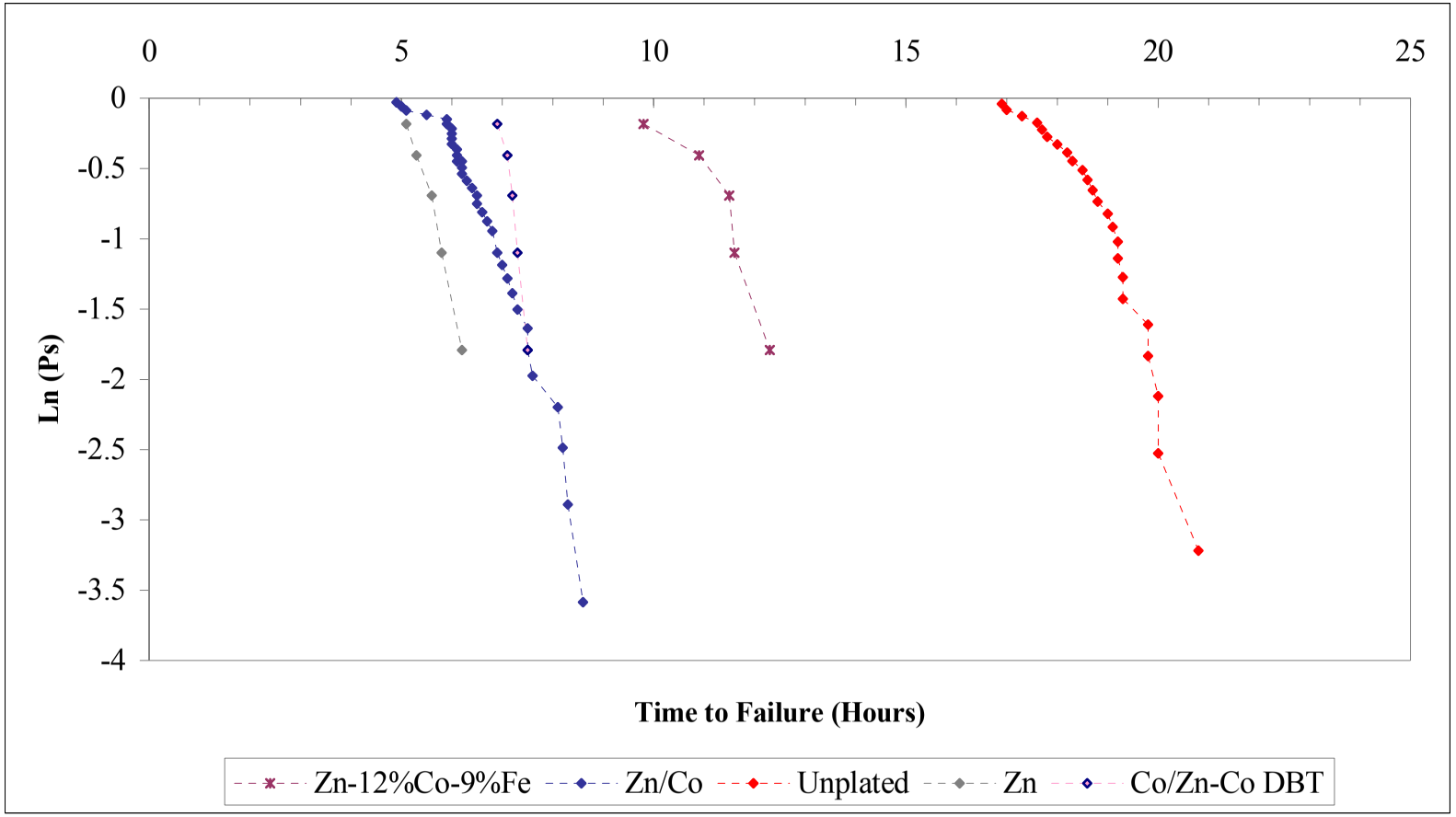


**Fig 8 Weibull plot comparing times to failure of specimens plated with zinc-cobalt-iron, zinc and zinc-cobalt**





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# Hydrogen embrittlement of high strength steel electroplated with zincÃ¢Â Â cobalt allo

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