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MATERIALS REPORT 117

HYDROGEN EMBRITTLEMENT OF CADMIUM-PLATED ULTRA-HIGH STRENGTH STEELS IN PAINT STRIPPERS

by

W. J. POLLOCK

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SUMMARY

Slow strain rate and electrochemical tests were applied to elucidate the mechanism of hydrogen embrittlement of high strength 4340 steel by paint strippers. Results show that hydrogen embrittlement does not occur unless the steel is cadmium plated and is due to hydrogen generation produced by the establishment of a galvanic couple between the steel and the cadmium in the paint stripper. The amount of embrittlement is seen to increase as the galvanic potential becomes increasingly more negative compared with the potential for the reduction of water to produce hydrogen. The role of inhibitors in the prevention of hydrogen embrittlement is discussed.

Additional keywords: paint removers; galvanic corrosion; cadmium coatings; aircraf; Australia, [illegible]

POSTAL ADDRESS: Director, Aeronautical Research Laboratories,
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1. INTRODUCTION

The use of ultra-high strength steel components in the aircraft industry has led to the development of many plating and paint systems designed to protect the steel from environmental attack. Since in-service deterioration and damage often leads to repair or replacement of these protective schemes, a large variety of paint strippers have been developed to remove paint and epoxy coatings without damaging the underlying metal. Due to the susceptibility of high strength steels to hydrogen embrittlement, it is important that hydrogen is not generated during exposure to the paint strippers as premature failure can subsequently occur without any sign of prior structural damage.

In the present work, slow strain rate and electrochemical techniques were used to investigate the hydrogen embrittlement susceptibility of 4340 steel to two phenolic (A, B) and four non-phenolic (C, D, E, F) paint strippers. The paint strippers had been previously tested in triplicate using notched C-ring specimens loaded to 75% of the notch tensile strength, immersed in the paint stripper for 60 s and left to hang for 100 h. All specimens exposed to paint strippers B and C failed within the 100 h period whereas the remaining specimens exposed to paint strippers A, D, E and F survived the 100 h test (1). Paint strippers that pass this test are considered 'non-embrittling'.

2. EXPERIMENTAL

The present work was carried out using round 4340 steel bar of composition 0·43 C, 0·85 Cr, 0·84 Mn, 0·25 Mo, 1·78 Ni, <0·01 P, 0·29 Si, 0·009 S, <0·05 Al, remainder Fe which complied with the US military specification MIL-S-5000E (2). Slow strain rate tests were conducted using notched tensile specimens that complied with the ASTM-F-519-77 specification (3) for hydrogen embrittlement testing (Fig. 1). The specimens were smaller than those defined in the standard specification but were considered acceptable within the terms of the specification by keeping the notch stress concentration factor within the range 2·9–3·3. Specimen blanks were machined prior to heat treatment and the specimen notch inserted by low-stress crush grinding after heat treatment. Specimen heat treatment involved austenitising at 815°C for 1 h, quenching into oil at 40–60°C, and double tempering at 260°C for 1 + 1 h. Specimens were cadmium plated to a thickness of 15 μm in a low-embrittlement bath complying with the Douglas Process Standard DPS 9·28 (4). After baking at 190°C/23 h, a number of specimens were also chromate passivated according to the Australian Defence Specification DEF (AUST)-110 (5). Specimens were then stored in a dry environment prior to conducting slow-strain rate tests in the paint stripper.

Slow strain rate tests were conducted using a 20 kN hard-beam tensile testing machine with a variety of gears enabling experiments to be conducted over a range of crosshead-displacement rates varying from 10^{-2} to 10^{-6} mm/s. Most tests were performed in triplicate and the mean fracture stress used as a parameter for quantifying the susceptibility to hydrogen embrittlement. A 40 ml teflon chamber was used to expose the central 25 mm portion of the specimen to the paint stripper (Fig. 2). A leak-tight seal was obtained by inserting the specimen into a hole in a teflon plug which in turn was screwed into a tapered threaded hole in the base of the environmental chamber.

Electrochemical measurements were conducted using 7 mm diameter cylindrical steel and cadmium specimens mounted to give a tight fit in a teflon holder. After the bare steel and cadmium specimens were polished with 600 grit silicon carbide paper, their open circuit potentials and the galvanic potential between them were measured in the paint strippers with respect to a saturated calomel electrode using a high impedance voltage follower. The pH of each paint stripper was also determined.

The morphologies of the steel fracture surfaces and the cadmium plate were studied in the scanning electron microscope.

3. RESULTS

The mean notch tensile strength of ten unembrittled tensile specimens tested in air at a crosshead-displacement rate of 2×10^{-4} mm/s was 2464 MPa with a standard deviation of 57 MPa. The mean fracture stress of six cadmium plated-and-baked specimens tested in air at the same crosshead-displacement rate was 2407 MPa with a standard deviation of 105 MPa. All specimens failed by transgranular microvoid coalescence and displayed no evidence of intergranular fracture.

Slow-strain rate experiments with cadmium plated-and-baked specimens in paint strippers A and B showed that the severity of embrittlement increased with decreasing applied crosshead-displacement rate (Fig. 3). At a crosshead-displacement rate of 2×10^{-3} mm/s, neither paint stripper showed signs of embrittling the steel, whereas at 2×10^{-6} mm/s both paint strippers caused fracture to occur at 30–40% of the unembrittled fracture stress. Since the maximum difference in fracture stress between the two paint strippers occurred at a crosshead-displacement rate of 2×10^{-4} mm/s, all subsequent experiments were conducted at this rate of testing. A comparison of the mean fracture stress of specimens tested in the six paint strippers at a crosshead-displacement rate of 2×10^{-4} mm/s revealed that the two paint strippers causing failure in the notched C-ring test also produced the lowest mean fracture stress in the slow-strain rate test (Fig. 4). No significant difference in results was obtained in tests with chromate-passivated and non-passivated cadmium-plated specimens. All embrittled specimens showed evidence of intergranular fracture with the proportion increasing with decrease in measured fracture stress.

Slow strain rate experiments with bare steel tensile specimens tested in either paint stripper A or B showed no evidence of embrittlement (Fig. 5). Further tests showed that the establishment of a galvanic couple between the bare steel specimen and a piece of cadmium of equivalent area immersed in the paint stripper B during straining resulted in premature failure (Fig. 5). Furthermore, exposure of a cadmium plated-and-baked specimen to paint stripper B for a week resulted in premature failure when the specimen was subsequently cleaned and tested in air (Fig. 5). Similar tests with paint stripper A showed that the degree of embrittlement produced by galvanic coupling was less than with paint stripper B.

The open circuit potentials of steel and cadmium and the galvanic potential of the steel/cadmium couple in the various paint strippers are listed in Table 1 along with the pH values of the six paint strippers. The time taken to reach a steady-state galvanic potential was short ($\ll 1$ h) for paint strippers B, C, D and E whereas many hours were required before a steady-state value was reached with the remaining two paint strippers. It is believed that the kinetics of the various reactions that take place at the cadmium and steel surface would depend partly on the viscosity of the paint stripper and the effect of any added inhibitor designed to prevent corrosion.

4. DISCUSSION

The electrochemical studies provide the necessary clues to explain the hydrogen embrittlement of cadmium plated-and-baked 4340 steel in paint strippers. In mildly alkaline media, adsorbed hydrogen atoms are generated at the steel surface by electrochemical reduction of water (6):

$$H_2O + e^- \rightarrow H_{ad} + OH^- \qquad (1)$$

The absorbed hydrogen atoms can then combine either chemically or electrochemically according to equations (2) and (3) respectively to form hydrogen gas (6):

$$2H_{ad} \rightarrow H_2 \qquad (2)$$

$$H_{ad} + H_2O + e^- \rightarrow H_2 + OH^- \qquad (3)$$

Alternatively, the adsorbed hydrogen atoms can be absorbed into the steel:

$$H_{ad} \rightarrow H_{ab} \qquad (4)$$

In mildly alkaline paint strippers, cathodic reduction of water can only proceed at potentials more negative than the reversible hydrogen electrode potential (7) (Fig. 6). Since the open circuit potential of 4340 steel in all the paint strippers is significantly more positive than the potential for cathodic-reduction of water (Fig. 6), embrittlement is not expected and this is confirmed using slow-strain rate testing of bare steel specimens (Fig. 5). Embrittlement of cadmium-plated specimens is shown to be related to the galvanic potential in the paint strippers. Since the potential for the cathodic reduction of water (E_H) depends on pH, E_H is subtracted from the galvanic potential (E_{galv}) for each paint stripper and then compared with the results of the slow strain rate tests (Fig. 7). The galvanic potentials of the two most embrittling paint strippers (B, C) are more negative than the corresponding E_H values, whereas the reverse is true for the other four paint strippers. This method could therefore be used to make an inexpensive, rapid preliminary assessment of the hydrogen embrittlement susceptibility of a particular paint stripper. It is suggested that paint strippers producing values of $E_{galv}-E_H < 50$ mV be considered likely candidates for generating significant embrittlement in cadmium-plated high-strength steels.

Galvanic coupling between the cadmium and steel in cadmium-plated specimens can occur only if the environment can penetrate the thin cadmium layer (12–15 μm deep). The highly porous nature of the cadmium deposited in the low-embrittlement bath ensures that the environment can penetrate to the steel surface (Fig. 8). The galvanic couple existing on cadmium-plated specimens immersed in paint strippers comprises a large anodic area (cadmium) coupled to a small cathodic area (steel) (Fig. 9). Since the anodic and cathodic currents must be equal, the large anodic area provides the driving force for high cathodic current densities at the small areas of steel exposed to the paint stripper. The mechanism is analogous to pitting, crevice corrosion and galvanic corrosion in situations where the cathodic and anodic areas are reversed and where oxygen reduction at the large cathode area provides the driving force for localized corrosion in the pit or crevice. The present situation might be considered potentially more dangerous since the localized cathodic hydrogen charging of the steel can proceed without any external signs of attack. In addition, since cadmium is particularly effective in preventing the escape of hydrogen from the steel at room temperature, repeated applications or incomplete removal of the paint stripper could lead to a cumulative build-up of hydrogen within the steel and cause potentially dangerous situations to develop during the life of a high strength steel component. In situations where cadmium-plated components are periodically subjected to environments where detrimental galvanic coupling is suspected, baking at 190°C for 23 h should be sufficient to eliminate any risk of failure by hydrogen embrittlement.

Many organic-based adsorption inhibitors have been found to be successful in minimizing corrosion of steel in acids (8–13). Hydrogen embrittlement can also be reduced by either curtailing reactions 1 and 4 and/or promoting reactions 2 and 3. The extent to which adsorbed ions can catalyze these reactions depends on the steel, the acid, the nature of the inhibitor and its concentration. Certain quaternary ammonium salts have been found to be useful in reducing corrosion and hydrogen entry into steel in acid solution (12, 14). Although little work has been done to investigate the behaviour of quaternary ammonium salts in neutral or mildly alkaline solutions, it was considered worthwhile to investigate the potential of some quaternary ammonium salts in inhibiting hydrogen embrittlement of 4340 steel in embrittling paint strippers B and C. Although small additions (0·1% wt.) of three quaternary salts, Dodigen 5594 (soya bean alkyl trimethyl ammonium chloride), Preventol R90 (C_{12}/C_{14} benzyl dimethyl ammonium chloride) and Methylene Blue did not increase the fracture stress of cadmium plated-and-baked specimens, larger quantities (2% wt.) of Methylene Blue were found to be particularly effective in both paint strippers (Fig. 4). It has been suggested that the Methylene Blue cation is preferentiallly reduced thereby suppressing the reduction of water and the subsequent adsorption of hydrogen on the steel surface (4).

An alternative approach that might prove worthwhile is to find an inhibitor that would raise the open circuit potential of the cadmium to a value above the critical potential for the reduction of water. This approach should be feasible in alkaline media bearing in mind that the standard electrode potential for the reaction $Cd^{2+}+2e^{-} \rightarrow Cd$ is $-0{\cdot}64$ V (sce).

4. CONCLUSIONS

Both electrochemical and slow-strain rate testing have proved to be versatile tools in studying hydrogen embrittlement of 4340 steel by paint strippers. Results show that the galvanic action between the cadmium and the steel can result in embrittlement of cadmium-plated steels and that embrittlement can be avoided by ensuring that the galvanic potential between the steel and cadmium is kept substantially more positive than the potential for the reduction of water to generate hydrogen. It is hoped that this approach will lead to improved performance of paint strippers thereby removing the risk of failure of components by hydrogen embrittlement during service.

ACKNOWLEDGEMENTS

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TABLE 1

Electrochemical Measurements in Paint Strippers

	Paint Stripper					
	A	B	C	D	E	F
Open circuit potential of 4340 Steel (V_{sce})	−0·33	−0·63	−0·41	−0·25	−0·41	−0·41
Open circuit potential of cadmium (V_{sce})	−0·75	−0·84	−0·94	−0·74	−0·64	−0·81
Steel/cadmium galvanic potential after 1 h (V_{sce})	−0·61	−0·75	−0·93	−0·68	−0·48	−0·69
Steady-state galvanic potential (V_{sce})	−0·74	−0·75	−0·93	−0·69	−0·49	−0·61
pH	9·8	8·0	11·3	8·6	8·8	11·5

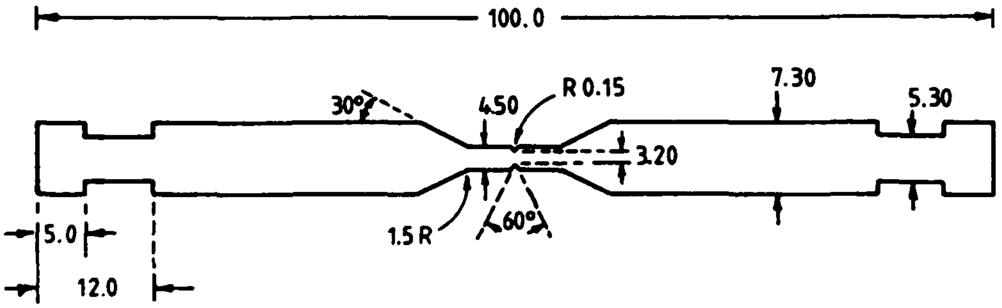

FIG. 1 4340 STEEL NOTCHED TENSILE SPECIMEN. DIMENSIONS IN mm.

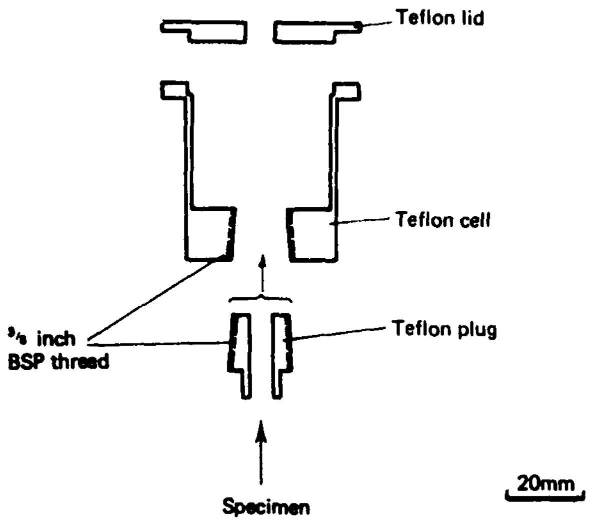

FIG. 2 ENVIRONMENTAL CELL FOR TESTING NOTCHED STEEL SPECIMENS IN PAINT STRIPPERS.

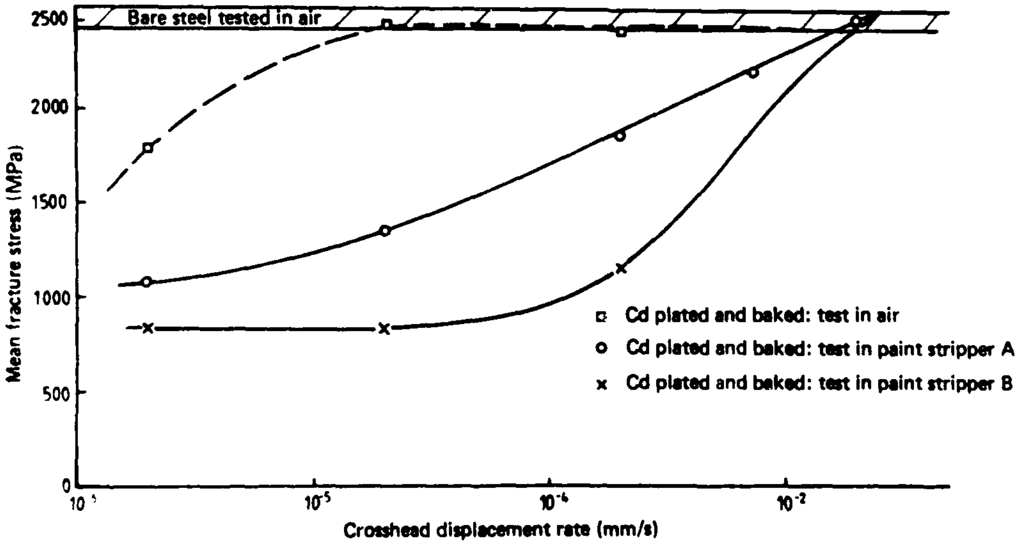

FIG. 3 FRACTURE STRESS OF CADIUM PLATED-AND-BAKED 4340 STEEL SPECIMENS TESTED IN PAINT STRIPPERS A AND B AT VARIOUS CROSSHEAD-DISPLACEMENT RATES.

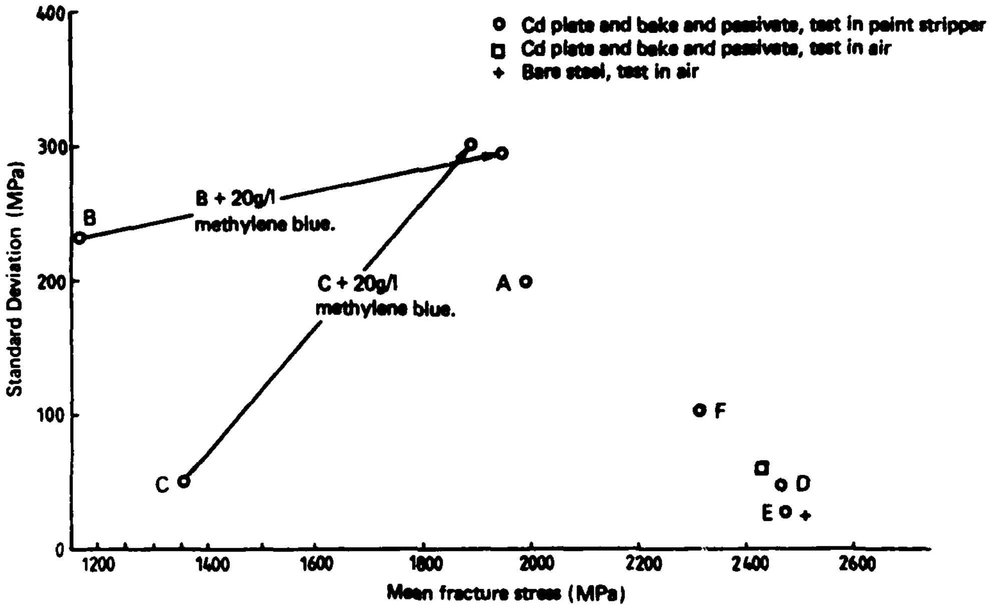

FIG. 4 MEAN FRACTURE STRESS AND STANDARD DEVIATION OF CADMIUM PLATED-AND-BAKED 4340 STEEL SPECIMENS TESTED IN VARIOUS PAINT STRIPPERS AT A CROSSHEAD-DISPLACEMENT RATE OF 2 X 10^{-4} mm/s.

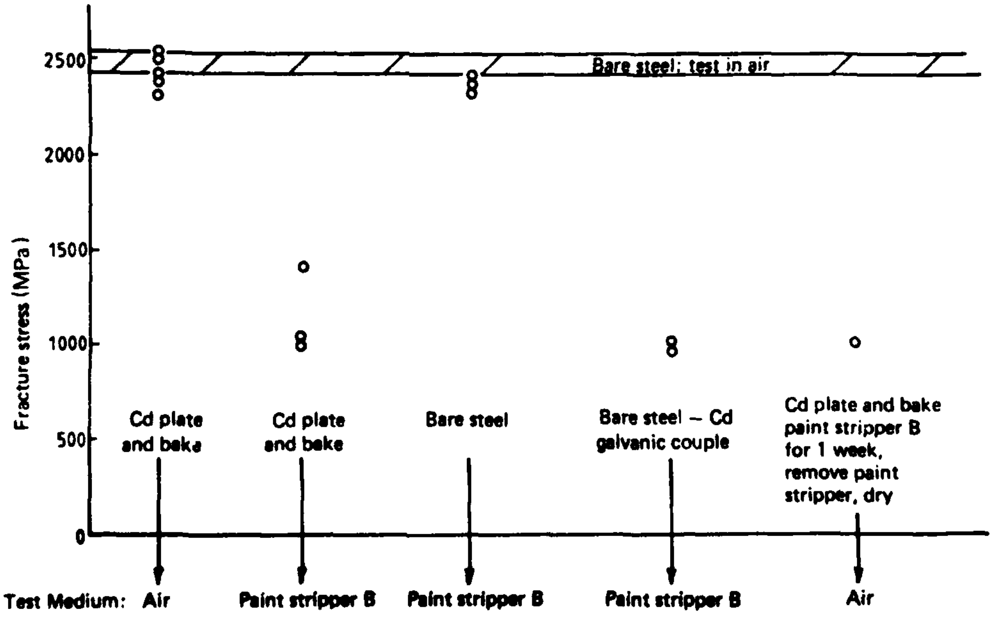

FIG. 5 FRACTURE STRESS OF 4340 STEEL SPECIMENS TESTED AT A CROSSHEAD-DISPLACEMENT RATE OF 2 X 10^{-4}mm/s.

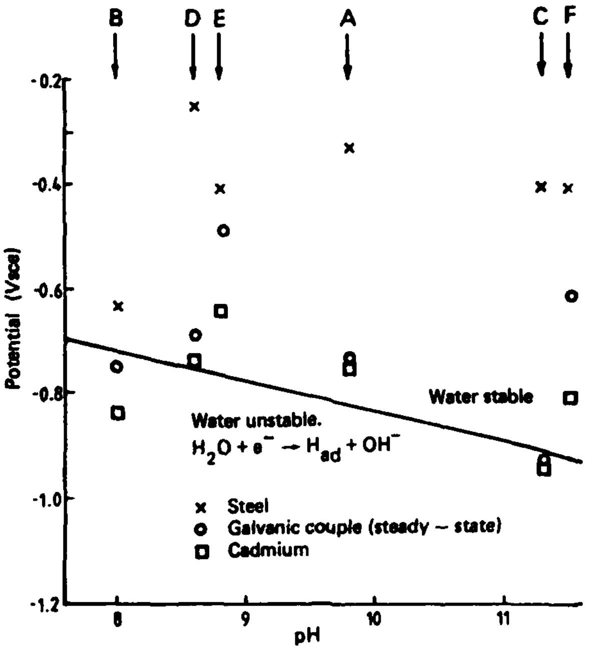

FIG. 6 POURBAIX DIAGRAM FOR STEEL AND CADMIUM IN PAINT STRIPPERS.

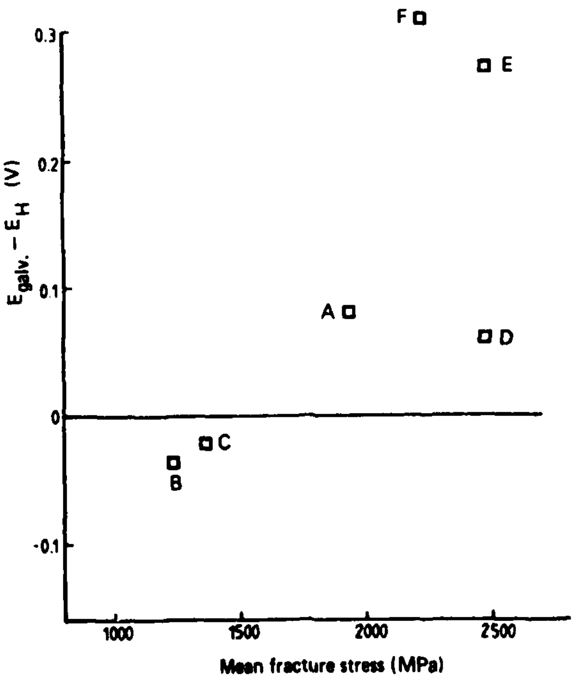

FIG. 7 RELATIONSHIP BETWEEN $E_{galv} - E_H$ AND FRACTURE STRESS.

FIG. 8 SCANNING-ELECTRON MICROGRAPH OF POROUS CADMIUM LAYER DEPOSITED IN A LOW-EMBRITTLEMENT PLATING BATH.

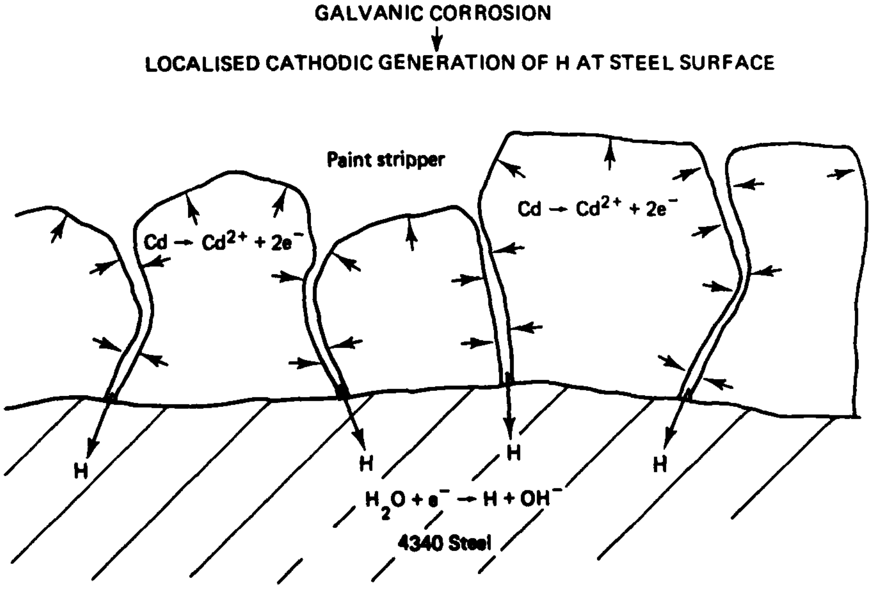

FIG. 9 LOCALISED CATHODIC GENERATION OF HYDROGEN AT STEEL SURFACE IN CADMIUM PLATED-AND-BAKED SPECIMENS IMMERSED IN PAINT STRIPPERS.

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