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AERONAUTICAL RESEARCH LABORATORY

MELBOURNE, VICTORIA

Aircraft Materials Report 122

STATISTICAL TREATMENT OF SLOW STRAIN RATE DATA FOR ASSESSMENT OF HYDROGEN EMBRITTLEMENT IN LOW ALLOY HIGH STRENGTH STEEL

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SUMMARY

Slow strain rate testing has been used to quantify the degree of hydrogen embrittlement produced in high strength 4340 steel by plating processes and aircraft maintenance chemicals. The results of slow strain rate tests, conducted at a crosshead displacement rate of 2 x ~~10^{-4}~~ mm/s using samples of three notched tension specimens in various paint strippers, show that a mean fracture stress of 1850 MPa can be correlated with the pass/fail criterion for acceptability of paint strippers in an existing Standard Notched C-ring Test. Statistical analysis of the slow strain rate data allows criteria to be established which will ensure a 99% probability of identifying all paint strippers that fail the Notched C-ring Test. This analysis is also used to grade a series of products and processes in terms of their tendency to cause hydrogen embrittlement, and to identify environmental parameters that cause excessive scatter in hydrogen embrittlement tests. Criteria are also specified for acceptability of each heat treatment batch of 4340 steel specimens to ensure reproducibility of results obtained from slow strain rate tests.

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TABLE OF CONTENTS

TABLES 1 & 2

FIGURES 1 - 11

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DOCUMENT CONTROL DATA

1. INTRODUCTION

Many critical load-bearing components in both commercial and military aircraft are manufactured from low alloy high strength steel. Since this type of steel is particularly susceptible to hydrogen embrittlement, extreme care must be exercised during all manufacturing and maintenance procedures to avoid hydrogen generation and absorption into components. Many Standard Specifications have been developed to screen products[1-4] (paint strippers, washing solutions, solvents) and processes[1, 5, 6] (electroplating) using specimens manufactured from high strength air-melted 4340 steel, known for its high susceptibility to hydrogen embrittlement. In these tests, three or four specimens are loaded to a fixed percentage of the unembrittled fracture stress and held for specified periods up to 200 hours. Failure or visual evidence of hydrogen cracking in one or more specimens is used as a basis for rejection of a given process or product. Unfortunately, high scatter frequently occurs in hydrogen embrittlement tests, and the small number of specimens normally utilised in current Standard Methods does not generate sufficient data for the reliability of products and processes to be analysed in a statistically rigorous manner. Moreover, Standard Methods are also unable to grade a range of products or processes in terms of their tendency to cause embrittlement when no specimens fail during the time scale of the test. The importance of this capability becomes crucial in cases where intermittent exposure of components to a variety of maintenance environments can cause hydrogen to build up over long periods of time thereby leading to premature delayed failure during service. Alternative methods should therefore be sought with a capability to identify the safest product or process for a particular application, and to validate this choice by rigorous statistical analysis.

Slow strain rate testing is frequently used to study environmentally assisted fracture such that results can be achieved more rapidly than conventional constant load tests.[7] In previous work[8-11], it was possible to characterise the degree of hydrogen embrittlement produced by a range of aircraft maintenance processes by analysis of the mean fracture stress and standard deviation produced by loading high strength 4340 steel notched tensile specimens to failure at a constant crosshead displacement rate. The present paper discusses how the results of these slow strain rate tests can be analysed statistically to overcome many of the limitations pertaining to existing Standard Hydrogen Embrittlement Tests.

2. EXPERIMENTAL

Notched tension specimens, compatible with the reduced load requirements for the type 1a specimen in Standard ASTM F519-77[1], were manufactured using three separate lots of aircraft quality air-melted 4340 steel which complied with the MIL-S-5000E Specification[7] (Fig. 1). Heat treatment of eight separate specimen batches involved austenitisation at 860°C for 1 hour, oil-quenching to 60°C and tempering twice at 260°C for 1 hour with air-cooling between tempers. The notch was prepared by low stress grinding after heat-treatment. Un-notched tension specimens with a gauge length of 12.7 mm and minimum diameter of 3.2 mm were also manufactured to determine the ultimate tensile strength of the steel.

Specimens were loaded to failure using a 20kN motor-driven hard-beam tension testing machine which had a variety of gears enabling tests to be conducted over a range of crosshead displacement rates varying from 10^{-2} to 10^{-6} mm/s. Unless otherwise stated, mean fracture stress and standard deviation values quoted in this paper were calculated from the results of testing three specimens in an identical manner.

Assessment of the degree of internal hydrogen embrittlement was undertaken by conducting slow strain rate tests at 20°C in air after either: a) cadmium (Cd) or Cd-Ti electroplating and baking, or b) exposure of unstressed plated specimens to deleterious maintenance products (eg. paint strippers) or environments (aqueous sodium chloride solutions). In a further series of tests, porous low hydrogen embrittlement (LHE) Cd plated-and-baked (190°C/23 hours) specimens were loaded to failure in the presence of a series of six proprietary paint strippers (A, B, C1, C2, D, E) to assess their tendency to cause hydrogen-assisted cracking. Samples C1 and C2 were two different batches of the same paint stripper and had the same nominal composition, but exhibited different tendencies to cause hydrogen-assisted cracking. Further details associated with the plating-and-baking procedures and paint strippers have been described elsewhere.[8-11]

The tendency of paint strippers to cause hydrogen-assisted cracking was also assessed using a Standard Notched C-ring Test.[8] Each test required three LHE Cd plated-and-baked specimens to be loaded in air to 75% of the notched bend strength and the notched region of the specimen immersed in the paint stripper. If two or more specimens failed within 100 hours, the environment was considered embrittling. If one specimen failed, a further three specimens were tested, and the paint stripper was rejected if one or more specimens failed or showed any evidence of cracking.

3. RESULTS

The results of testing 21 un-embrittled notched bare steel tension specimens in air produced a mean fracture stress and standard deviation of 2456 MPa and 52 MPa respectively, thereby confirming that the fracture stress was independent of crosshead displacement rate over the range 2×10^{-3} to 2×10^{-6} mm/s. Similar tests with un-notched specimens revealed an ultimate tensile strength of 1845 MPa.

Results of duplicate tests carried out in air after a variety of Cd plating-and-baking procedures showed that the slow strain rate technique can differentiate between a wide range of hydrogen-embrittling treatments (Fig. 2). Tests carried out at a crosshead displacement rate of 2×10^{-4} mm/s can characterise the severely embrittling properties of a bright Cd plating bath whereas crosshead displacements rates of 2×10^{-6} mm/s must be used to distinguish between the embrittling characteristics of LHE Cd and Cd-Ti plating-and-baking treatments.

The embrittling properties of the six paint strippers were characterised by conducting tests in triplicate in which LHE Cd plated-and-baked specimens were loaded to failure in the presence of each paint stripper (Fig. 3, Table 1). A comparison of mean fracture stress values revealed paint stripper B to be most embrittling, with paint strippers D and E exhibiting minimum tendency towards hydrogen assisted cracking. This result was confirmed in the Notched C-ring Tests where paint strippers A, C1, D and E could be classified as "non-embrittling" whereas paint strippers B and C2 could be labelled "embrittling" (Table 2).

Since batches C1 and C2 of paint stripper C were found to be "non-embrittling" and "embrittling" respectively, further slow strain rate and Notched C-ring Tests were carried out in various mixtures of paint strippers C1 and C2. Despite the high standard deviation associated with some of the results of slow strain rate tests in paint stripper mixtures, the mean fracture stress increased with greater proportions of C1 in the paint stripper mixture (Fig. 4). The paint stripper mixture containing 75% C1 just passed the Notched C-ring Test whereas all specimens tested in the mixture containing 65% C1 consistently failed within 50 hours of commencing the test (Fig. 4, Table 2). Consequently the pass/fail condition for the Notched C-ring Test is most closely simulated by the paint stripper mixture containing 75% C1 and the corresponding mean fracture in the slow strain rate test conducted at a crosshead displacement rate of 2×10^{-4} mm/s is therefore estimated to be 1850 MPa.

The limited amount of testing and variable scatter associated with the slow strain rate data in Fig. 4 does not permit a proper statistical assessment of the results. The overall significance of a minimum acceptable mean fracture stress and comparative assessments of a range of products becomes more meaningful when the statistics of slow strain rate results encompassing a much wider data base are analysed. Mean fracture stress and standard deviation data for all tests conducted in triplicate[8-11] show results displaying considerable scatter (Fig. 5). The data can be divided into two groups: an anomalous group in which four sets of data display much higher scatter than all the remaining results (Table 1), and a main group of data which display standard deviation values ranging from 100 to 450 MPa at intermediate mean fracture stresses, with much lower scatter evident at both low and high mean fracture stress. Since the main group of data is derived from tests encompassing many steel lots and heat treatment batches as well as a range of crosshead displacement rates in both air and in paint strippers, the variation in standard deviation with mean fracture stress is likely to depend on metallurgical factors associated with crack initiation for this particular strength level of air-melted 4340 steel in both the embrittled and unembrittled condition.

If the main body of results is assumed to reflect a population which characterises acceptable data produced during slow strain rate tests, a statistical analysis can be done which allows:

a) identification of processes and products causing excessive scatter in hydrogen embrittlement tests,

b) establishment of a minimum acceptable mean fracture stress guaranteeing to any predetermined probability the identification of all paint strippers that fail the existing Standard Notched C-ring Test,

c) a series of products or processes to be graded in terms of their tendency to cause hydrogen embrittlement, and

d) acceptability criteria to be established for different heat treatment batches of 4340 steel thereby ensuring reproducibility of all slow strain rate results.

4. STATISTICAL ANALYSIS OF RESULTS

4.1 Anomalous Data

Before discussing the statistical properties of the main group of slow strain rate data, an analysis is undertaken to discuss the significance of the four data points exhibiting standard deviation >500 MPa (Fig. 5, Table 1).

When comparing two normal populations, a hypothesis test, based on comparisons of their two variances, is used to determine whether the population parameters are the same to some preset level of significance.[13] This analysis involves a null and alternative hypothesis concerning the two populations with variances σ_1^2 and σ_2^2:

Null hypothesis:	$H_0 : \sigma_1^2 \leq \sigma_2^2$
Alternative hypothesis:	$H_a : \sigma_1^2 > \sigma_2^2$

When independent random samples of size n_1 and n_2 are drawn from normal populations of equal variance, the ratio of the sample variances ($F^* = s_1^2/s_2^2$) will possess a probability distribution in repeated sampling known as the F distribution. If F^* exceeds the critical values $F(n_1\text{-}1, n_2 - 2, \alpha)$ of the distribution at a given significance level α, there is a 100 $(1 - \alpha)$ % probability of the null hypothesis H_0 being correctly rejected and σ_1^2 is declared to be significantly greater than σ_2^2.

This analysis is used to compare the variance from the main group of data with equivalent values from the four tests showing excessively high scatter. The reference sample taken from the main group of data includes seven results (n_2 = 21 specimens) falling within the mean fracture stress range 1580-1780 MPa (Fig. 5, Table 1). This sample displays an approximately normal distribution with a standard deviation (s_2) of 231 MPa (Fig. 6) and is compared with the result of one test (n_1 = 3, s_1 = 625 MPa) where three Cd plated-and-baked specimens were exposed to paint stripper C2 for 100 hours, the paint stripper removed and the specimen loaded to failure in air at a crosshead displacement rate of 2 x 10^{-4} mm/s (Table 1). Analysis of the F statistic (α = 0.01) indicates a 99% probability that the two populations are significantly different (Fig. 7), thereby confirming excessively high scatter in the paint stripper test. In this case, variability in paint stripper viscosity and pore size in the Cd coating may be factors leading to local variations in galvanic cathodic generation of hydrogen at the steel surface. A similar trend was observed in a second test in which a mean fracture stress and standard deviation of 2018 and 436 MPa respectively were obtained after exposure for 100 hours to a paint stripper mixture containing 50% C1 and 50% C2 (Fig. 5). This analysis predicts that unacceptably high scatter could occur in an existing Standard Specification[3] (MIL-R-83936B) where specimens are exposed to the paint stripper for a period of four hours prior to removal of the paint stripper and conducting a constant load test in air.

The remaining three data points displaying a standard deviation >500 MPa were obtained in tests where LHE Cd plated-and-baked specimens were loaded to failure in the presence of paint strippers A, D and E respectively. When the collective fracture stress values ($n_1 = 9$, $s_1 = 527$ MPa) are compared with the seven sets of reference data ($n_2 = 21$, $s_2 = 231$ MPa), whose mean fracture stress falls within the range 1580 - 1780 MPa, application of the F test ($\alpha = 0.01$) indicates a 99% probability that the two populations are significantly different. In each case, samples of paint stripper had been removed from their original drum and stored for several days in tin-coated containers prior to conducting the slow strain rate tests. All other tests involving samples of paint stripper taken directly from their original drums proved to be satisfactory (Table 1), and the anomalous results are probably due to localised contamination of the paint strippers leading to variable cathodic generation of hydrogen during the slow strain rate test.

This analysis shows the slow strain rate test is capable of identifying processes which enhance scatter in hydrogen embrittlement tests.

4.2 Pass/Fail Criteria in Slow Strain Rate Tests

Analysis of the present slow strain rate results, obtained by testing LHE Cd plated-and-baked specimens tested in mixtures containing paint strippers C1 and C2 at a constant crosshead displacement rate of 2×10^{-4} mm/s, shows that a mean fracture stress of 1850 MPa provides a realistic estimate of the pass/fail condition in the Notched C-ring Test (Fig. 4, Tables 1 and 2). Due to the high scatter associated with some of the slow strain rate results, any reliable pass/fail criterion based on a minimum acceptable mean fracture stress must be based on a statistical assessment of the slow strain rate data. This analysis relies on comparing the mean fracture stress (μ_1) of a test population, derived from slow strain rate data for any paint stripper under investigation, with that of a reference population (μ_2) where parameters are defined by conditions corresponding to the pass/fail criterion in the Notched C-ring Test. The two populations are assumed to have a normal distribution and a common variance (σ^2).

If samples are drawn from the test and reference populations and have sizes n_1 and n_2, means $\bar{x}_1$ and $\bar{x}_2$ and standard deviations s_1 and s_2 respectively, then the test population is deemed to be significantly different from the reference population if the experimental value for the test statistic (t^*) > $t(n_1 + n_2 - 2, \alpha)$ where[13]:

$$t^* = \frac{(\bar{x}_1 - \bar{x}_2) - (\mu_1 - \mu_2)}{s_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \tag{1}$$

and s_p is a pooled estimate for the standard deviation (σ) which is given by:

$$s_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}} \tag{2}$$

Theoretical values for $t(n_1 + n_2 - 2, \alpha)$ are derived from a table of critical values for Student's t distribution.

The reference set of data, which approximates the fracture stress population distribution in the region of the Notched C-ring pass/fail condition, is assumed to have a mean fracture stress ($\bar{x}_2$) of 1850 MPa and a standard deviation (s_2) of 241 MPa. The latter value was derived from statistical analysis of eight sets of data ($n_2 = 24$ specimens) whose mean fracture stresses fall in the range 1700 - 1850 MPa. If a standard deviation (s_1) of 350 MPa is assumed to be the worst possible case for any sample of 3 ($= n_1$) specimens tested in the paint stripper under investigation, then critical values of the mean fracture stress ($\bar{x}_1$) can be calculated above which the two populations are deemed to be significantly different (Fig. 8). Provided this criterion prevails, identification of a paint stripper which fails the notched C-ring test can be guaranteed to a probability level of at least 100 (1 - α) %. Choice of $\alpha = 0.01$ leads to a minimum acceptable mean fracture stress of 2234 MPa. This stringent requirement is essential to minimise the risk of dubious products or processes being used during maintenance since concentrations of hydrogen in steel as low as one part per million lead to delayed failure of critical load-bearing components.

This analysis illustrates how the concept of a minimum acceptable mean fracture stress can be used to reject deleterious paint strippers in slow strain rate tests which can be accomplished with three specimens in a maximum time of only 2.5 hours. The accuracy of the present analysis is dependent on the precision of the reference set of data describing the statistical behaviour of slow strain rate tests at a mean fracture stress equivalent to the pass/fail condition in the existing Notched C-ring Test. In the present work, the value for $\bar{x}_2$ = 1850 MPa was determined from a limited set of experimental data and a precise estimate of both $\bar{x}_2$ and s_2 requires more extensive slow strain rate and Notched C-ring Tests in the vicinity of the pass/fail result. Furthermore, values of $\bar{x}_2$ will vary with the pass/fail criterion in the Notched C-ring Test. If the rejection criterion for the Standard Notched C-ring Test is based on one failure out of three specimens tested, the equivalent slow strain rate mean fracture stress ($\bar{x}_2$) would be expected to exceed 1854 MPa.

Further statistical calculations based on a minimum acceptable mean fracture stress of 2234 MPa indicate a 93% probability of paint stripper A and a 50% probability of paint stripper C1 being rejected in a slow strain rate test using three specimens. The need for this stringent requirement is essential as more extensive Notched C-ring Tests[14] using paint stripper A have identified one failure out of ten specimens tested in two separate series of tests with different batches of paint stripper. This result confirms that hydrogen is generated during exposure of paint strippers that had been previously regarded as safe. If repeated exposure of components to marginally safe products result in a cumulative build-up of hydrogen in the steel, application of more stringent specifications would reduce the probability of delayed failure during their lifetime.

A similar analysis is also possible for assessment of internal hydrogen embrittlement caused by electrochemical plating processes. Most Standard Specifications require three or four specimens to be loaded to 75% of the unembrittled fracture stress and held in air for 200 hours. This is a more stringent requirement than is detailed in the present work and may result in higher values for $\bar{x}_2$. Determination of a minimum acceptable mean fracture stress for plating-and-baking processes would be extremely worthwhile financially since the reliability of a given plating bath could be determined in a maximum time of only 2.5 hours compared with one week for most existing Standard Methods and two-three weeks if repetition of tests is required as detailed in the Standard Method ASTM F519-77. Immediate decisions concerning the reliability of the plating bath would be made thereby preventing expensive delays in lost production or unnecessary grounding of aircraft. Concern has also been expressed with Standard Methods where repetition of tests is required.[1] Due to the delay period of approximately one week between plating the initial and second specimen batches, the characteristics of the bath could change and the second test may not be a genuine repeat of the first test. This problem would be circumvented by adoption of statistical criteria for slow strain rate testing.

4.3 Comparative Assessment of a Range of Products or Processes

The results of slow strain rate tests conducted with LHE Cd plated-and-baked specimens in a range of paint strippers at a crosshead displacement rate of 2 x 10^{-4} mm/s showed that the paint strippers could be graded in terms of their tendency to cause hydrogen-assisted cracking (Fig. 3). No discussion has been presented concerning the significance of these comparisons. Inferences concerning the difference between the mean fracture stress values obtained from tests in the various paint strippers can be determined using an analysis based on Student's t distribution. Since valid comparisons between two products are less critical than pass/fail criteria, comparisons are made on the basis of being able to distinguish between two paint strippers on their tendency to cause hydrogen-assisted cracking to a probability of 80% ($\alpha = 0.2$). For two paint stripper samples, each containing three specimens, the required interval for a significant difference in mean fracture stress ($\bar{x}_1 - \bar{x}_2$) is derived from equation 1 to give the results in Fig. 9 where the standard deviation for each sample has the same value. A significant difference is seen to exist between two products when the difference in mean fracture stress is approximately 1.25 times the standard deviation for the two tests. This value is substantially reduced if results are obtained with samples of 6 specimens (Fig. 9). If this analysis is applied to assess the results of specimens tested in various paint strippers at a crosshead displacement rate of 2 x 10^{-4} mm/s (Fig. 3), grading of all paint strippers except D with E and B with C2 is possible. Further testing of paint stripper D and E at a lower crosshead displacement rate would reveal which paint stripper shows the least tendency to hydrogen-assisted cracking. Although samples of 3 specimens are insufficient to distinguish between the populations representing paint strippers B and C2, repetition of the analysis using data for paint stripper C2 in Table 1 indicates that paint stripper B is significantly more embrittling than paint stripper C2.

4.4 Reproducibility of Specimen Batches

The current program utilised three separate lots of 4340 steel to manufacture notched tension specimens which were subsequently heat-treated in eight separate batches. Since the sole purpose of this work is characterisation of the embrittling tendencies of various products and processes, it is essential to ensure that no variation in properties occurs between different batches of 4340 steel specimens. In this work all steel lots complied with the MIL-S-5000E Specification and specimens manufactured from all heat treatment batches showed minimal difference in notched tensile strength. A large number of LHE Cd plated-and-baked specimens was tested in paint stripper C2 at a crosshead displacement rate of 2 x 10^{-4} mm/s since this product was found to be embrittling and gave reproducible results. Although a histogram of fracture stress values from 22 tests revealed a bimodal distribution (Fig. 10), the high mean fracture stress values were all found to originate from one particular heat treatment batch. Statistical comparison of the sample means indicates a 99% probability that the two sections of the distribution are significantly different. The reason for the two different sets of results has not been established and further work is required to assess:

a) possible variation in residual stress in the notch caused by heat-treatment and low stress grinding, and

b) effect of inclusion density and size on susceptibility to hydrogen-assisted cracking.

This example illustrates the necessity to screen every batch of specimens to ensure reproducibility of results. Use of a reproducibly embrittling environment may serve as an alternative to screening methods used in Standard Specifications where specimens are Cd plated in embrittling and non-embrittling baths to ensure that specimens will respond in a predicted manner.[1] The results in Fig. 10 provide a reference data base whereby an acceptability criterion can be established using Student's t distribution for any heat-treatment batch where six specimens are LHE Cd plated and baked prior to loading to failure in paint stripper C2 at a crosshead displacement rate of 2 x 10^{-4} mm/s (Fig. 11). If the mean fracture stress is outside the range 1377 ± 176 MPa, then Student's t distribution indicates a 95% probability that the population parameters of the steel batch are significantly different from those of the 7 batches of steel used to generate the results detailed in Fig. 10. If this criterion for acceptability of a given steel batch is adopted, then the slow strain rate mean fracture stress which equates to the pass/fail criterion in the Notched C-ring Test could be in error by an amount between 0 and ± 176 MPa. Although this value could be as high as 2026 MPa (ie. 1850 + 176 MPa), the minimum acceptable mean fracture stress of 2234 MPa derived in Section 5.2 would still ensure a 90% probability of rejecting any deleterious paint stripper. This acceptability criterion is still far more rigorous than those detailed in existing Standard Methods.

An alternative approach involves a more detailed evaluation of each specimen batch in which a large number of LHE Cd plated-and-baked specimens are tested in paint stripper C2. If the mean fracture stress exceeds 1377 MPa, then this difference could be added to the minimum acceptable fracture stress of 2234 MPa to ensure a 99% probability of detecting all paint strippers that fail the Standard Notched C-ring Test.

CONCLUSIONS

A successful Standard Method should not only provide a reliable assessment of properties under investigation, but should also prove to be economically viable. In the case of hydrogen embrittlement testing, the slow strain rate method can fulfil both these requirements. When applied to assess the tendency of various products and processes to induce hydrogen embrittlement in low alloy high strength steel, statistically significant results are obtained with a batch of 3 specimens in a maximum test time of only 2.5 hours. Statistical analysis of the results leads to the following conclusions:

a) Contamination of paint strippers and intermittent exposure of paint strippers to Cd plated specimens can cause unacceptably high scatter in subsequent hydrogen embrittlement tests,

b) For slow strain rate rests conducted in triplicate with LHE Cd plated-and-baked specimens which are loaded to failure at a crosshead displacement rate of 2×10^{-4} mm/s in the presence of a test paint stripper, a minimum acceptable mean fracture stress of 2234 MPa will ensure a 99% probability of identifying all products that fail the Standard Notched C-ring Test,

c) A range of products or processes can be graded to a probability level of 80% in terms of their tendency to cause hydrogen embrittlement when the mean fracture stress of specimens tested in triplicate differs by an amount greater than 1.3 times their standard deviation,

d) Acceptability criteria for different heat-treatment batches of 4340 steel specimens can be specified from the results of slow strain rate tests in a paint stripper with a well-defined susceptibility for causing hydrogen embrittlement.

The results presented in this paper provide a quantitative basis for estimating the degree of hydrogen embrittlement in low alloy high strength steel and it is hoped that adoption of such methods will lead to safer operation of aircraft structure. Furthermore, the same statistical treatment of slow strain rate data could also be used to assess the tendency of deleterious environments to cause delayed failure in other materials susceptible to environmentally assisted fracture.

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TABLE 1

Fracture Stress Data for Slow Strain Rate Tests

Acceptable Data

Test	No. of specimens	Fracture Stress (MPa)	Mean Fracture Stress (MPa)	Standard Deviation (MPa)
Paint Stripper A	11	1717, 1726, 1733, 1736, 1796, 1962 2117, 2127, 2157, 2174, 2327	1961	227
Paint Stripper B	3	1002, 1019, 1411	1144	231
Paint Stripper C1	3	2084, 2276, 2317	2226	124
75% C1 + 25% C2	3	1439, 2029, 2093	1854	360
65% C1 + 35% C2	3	1315, 1687, 2024	1675	355
50% C1 + 50% C2	3	1498, 1603, 1691	1596	99
25% C1 + 75% C2	3	1121, 1532, 1563	1405	246
Paint Stripper C2	17	1072, 1152, 1156, 1284, 1304, 1304 1336, 1355, 1356, 1390, 1401, 1426 1445, 1477, 1553, 1578, 1826	1377	178
Paint Stripper D	3	2414, 2477, 2506	2466	47
Paint Stipper E	3	2441, 2483, 2493	2472	28
All tests conducted in triplicate with mean fracture stress in range 1580-1780 MPa	21	1315, 1416, 1466, 1471, 1498, 1501 1603, 1608, 1616, 1625, 1682, 1687 1691, 1820, 1822, 1869, 2004, 2022 2024, 2044, 2077	1707	231
All tests conducted in triplicate with mean fracture stress in range 1700-1850 MPa	24	1446, 1471, 1554, 1579, 1582, 1595 1608, 1616, 1619, 1625, 1662, 1682 1799, 1810, 1820, 1822, 1869, 2004 2022, 2032, 2077, 2167, 2193, 2270	1789	241

Anomalous Data

Test	No. of specimens	Fracture Stress (MPa)	Mean Fracture Stress (MPa)	Standard Deviation (MPa)
Paint Stripper A	3	1126, 1380, 2174	1560	547
Paint Stripper D	3	1144, 1507, 2316	1656	600
Paint Stripper E	3	1155, 1539, 2438	1711	658
Anomalous steel batch/ Paint Stipper C2	5	2146, 2227, 2261, 2269, 2278	2236	54
100 hours exposure to paint stripper C2, test in air	3	1242, 1470, 2421	1711	625

TABLE 2

Results of notched C-ring tests in paint strippers - specimens loaded to 75% of notched breaking strength and held for 100 h

Paint Stripper	Time to Failure, h			Pass/Fail
	Specimen 1	Specimen 2	Specimen 3	
A	P	P	P	Pass
B	<1	<1	<1	Fail
C1	P	P	P	Pass
C2	<1	<1	1	Fail
D	P	P	P	Pass
E	P	P	P	Pass
50% C1 + 50% C2	*20*	*20*	*60*	Fail
65% C1 + 35% C2	24	24	48	Fail
75% C1 + 25% C2	P	P	60	Pass
75% C1 + 25% C2 (repeat)	P	P	P	

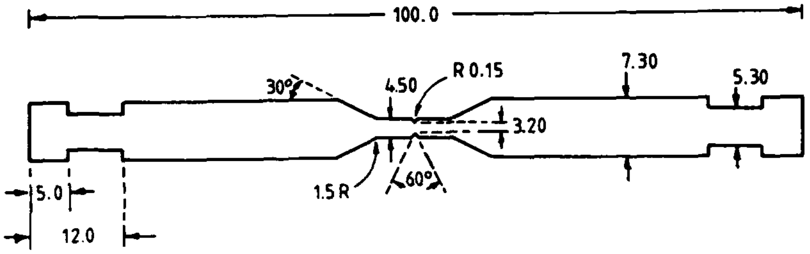

Figure 1: 4340 Steel Notched Tensile Specimen (dimensions in mm).

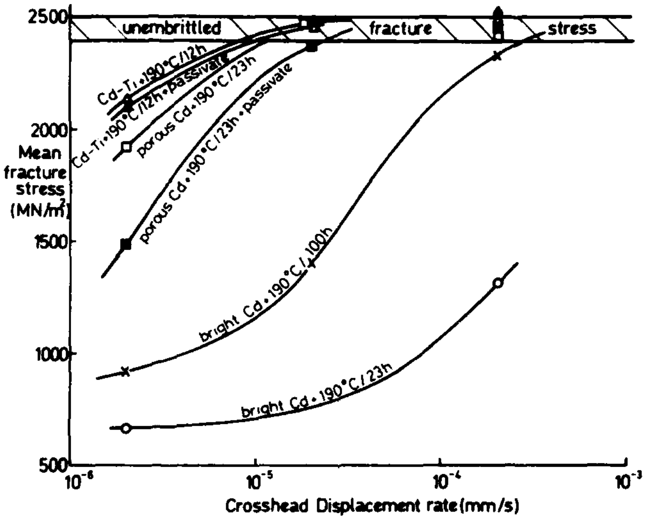

Figure 2: Mean fracture stress of Cd and Cd-Ti plated-and-baked 4340 steel specimens tested in air at various crosshead displacement rates.

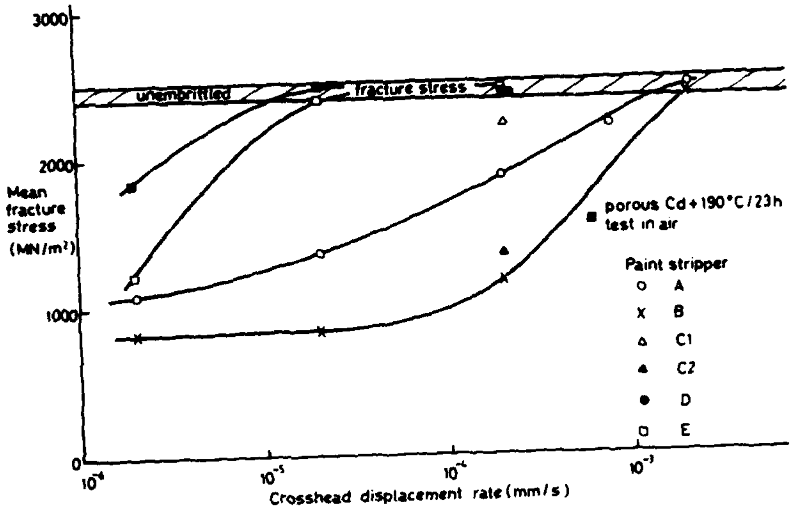

Figure 3: Mean fracture stress of porous Cd plated-and-baked 4340 steel specimens tested *in paint strippers at various crosshead displacement rates.*

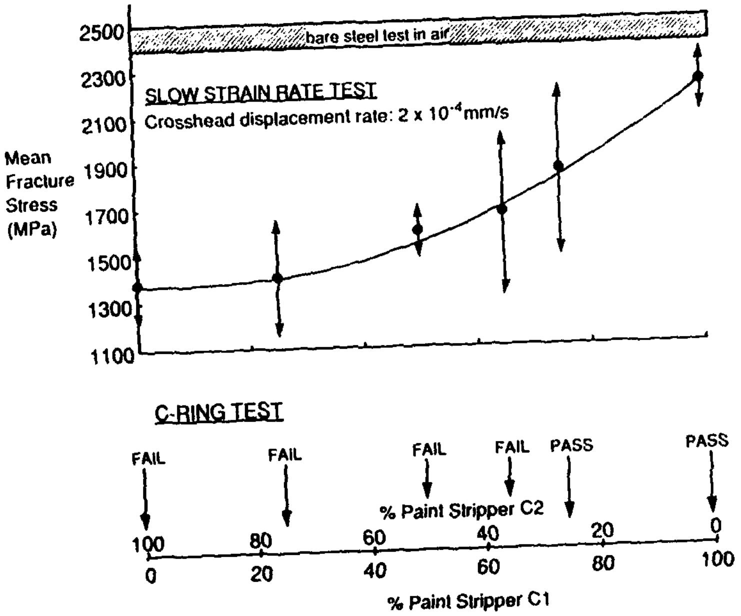

Figure 4: Results of notched C-ring and slow strain rate tests in mixtures of paint strippers C1 and C2 using LHE Cd plated specimens baked at 190°C/23 h. In slow strain rate data, arrows indicate mean fracture stress ± standard deviation.

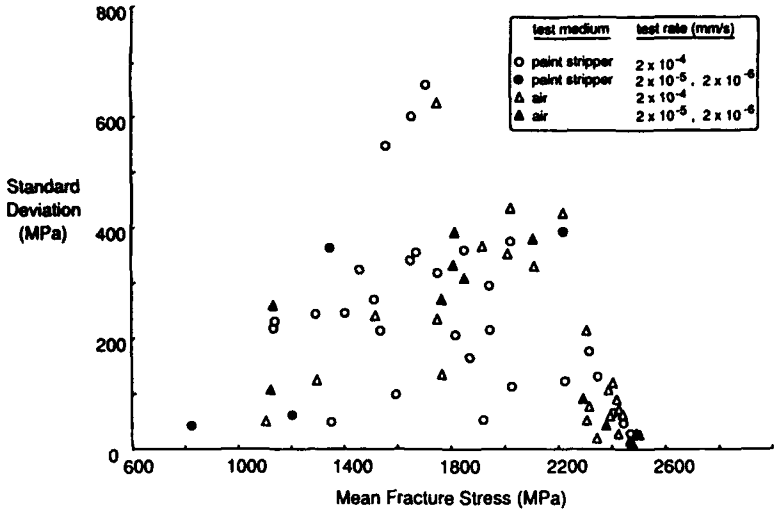

Figure 5: Mean fracture stress and standard deviation of all tests conducted in triplicate.

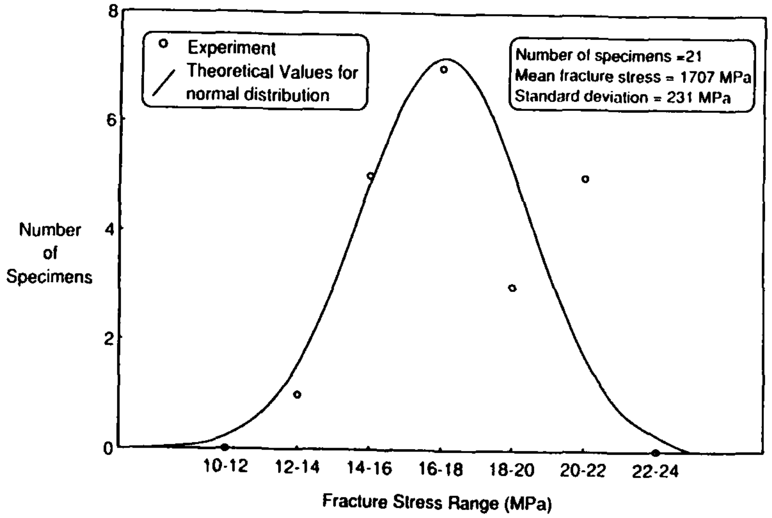

Figure 6: Frequency distribution of fracture stress data taken from seven sets of specimen tests conducted in triplicate whose mean fracture stress fall within the range 1580 to 1780 MPa.

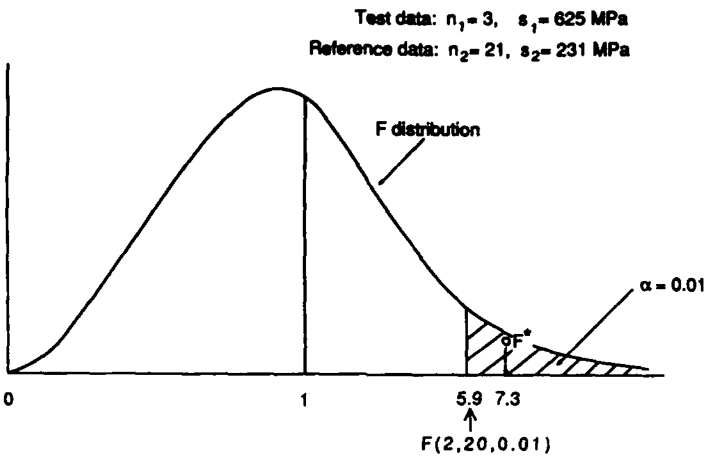

Figure 7: Application of F distribution test to compare reference and test data in slow strain rate tests.

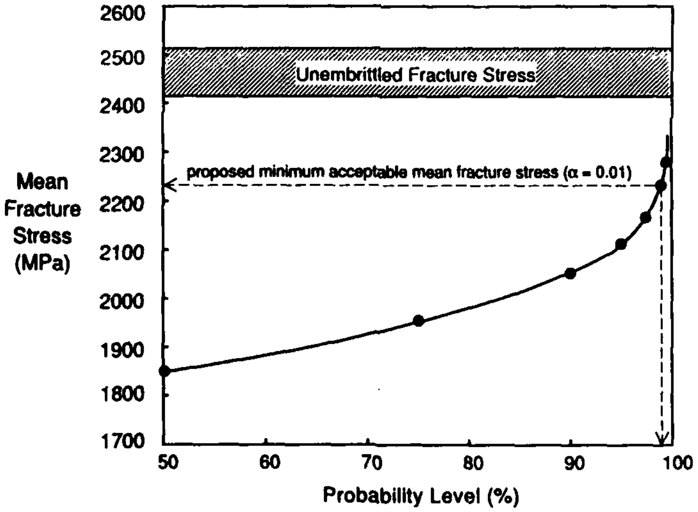

Figure 8: Values of mean fracture stress ($\bar{x}_1$), based on testing three slow strain rate specimens to failure in the presence of a test paint stripper ($n_1 = 3$, $s_1 = 350$ MPa), necessary to ensure a 100 (1 - α) % probability that the population parameters are significantly different from those of a reference population described by a set of data ($n_1 = 24$, $\bar{x}_2 = 1850$ MPa, $s_2 = 241$ MPa) which reflects the properties of the pass/fail conditions obtained in a Standard Notched C-ring test.

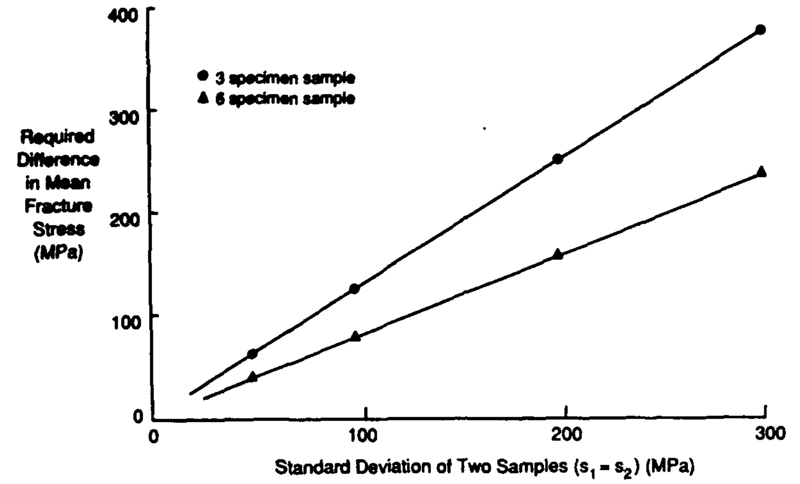

Figure 9: Difference in mean fracture stress necessary to distinguish between two paint strippers to a probability of 80% on their tendency to cause hydrogen-assisted cracking based on testing either three or six specimens to failure for different values of standard deviation.

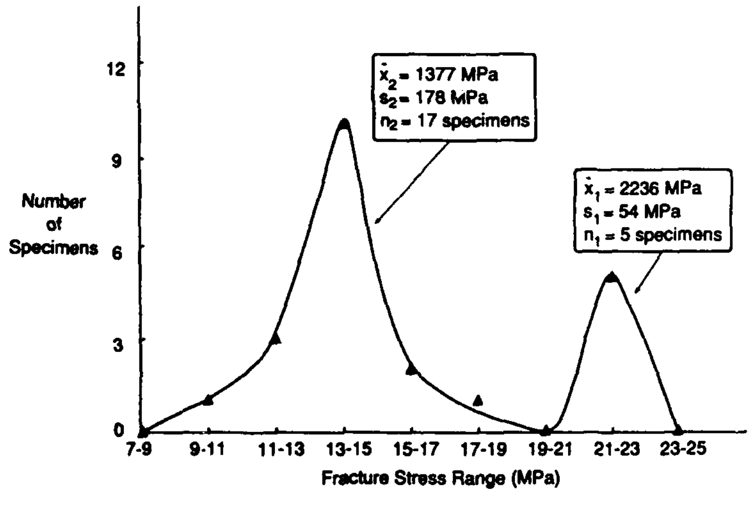

Figure 10: Frequency distribution of fracture stress data produced by testing 22 LHE Cd plated-and-baked specimens in paint stripper C2 at a crosshead displacement rate of 2 x 10^{-4} mm/s.

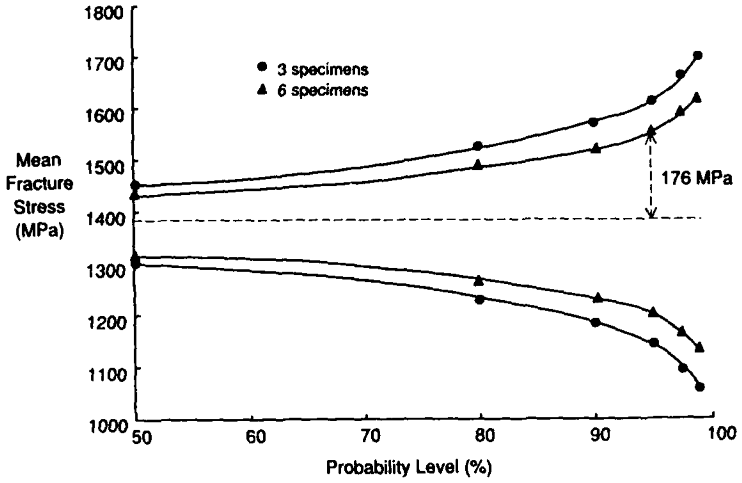

Figure 11: Range of mean fracture stress values within which the results of testing 3 or 6 LHE Cd plated-and-baked specimens in paint stripper C2 at a crosshead displacement rate of 2 x 10^{-4} must lie for their population parameters to be considered the same to any given probability level as those of a reference population described by the set of data (n_2 = 17, s_2 = 178 MPa, $\bar{x}_2$ = 1377 MPa).

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16. ABSTRACT

Slow strain rate testing has been used to quantify the degree of hydrogen embrittlement produced in high strength 4340 steel by plating processes and aircraft maintenance chemicals. The results of slow strain rate tests, conducted at a crosshead displacement rate of 2×10^{-4} *mm/s using samples of three notched tension specimens in various paint strippers, show that a mean fracture stress of 1850 MPa can be correlated with the pass/fail criterion for acceptability of paint strippers in an existing Standard Notched C-ring Test.*

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16. ABSTRACT (CONT).

Statistical analysis of the slow strain rate data allows criteria to be established which will ensure a 99% probability of identifying all paint strippers that fail the Notched C-ring Test. This analysis is also used to grade a series of products and processes in terms of their tendency to cause hydrogen embrittlement, and to identify environmental parameters that cause excessive scatter in hydrogen embrittlement tests. Criteria are also specified for acceptability of each heat treatment batch of 4340 steel specimens to ensure reproducibility of results obtained from slow strain rate tests.

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