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SLOW STRAIN RATE TESTING OF HIGH STRENGTH LOW-ALLOY STEELS : A TECHNIQUE FOR ASSESSING THE DEGREE OF HYDROGEN EMBRITTLEMENT PRODUCED BY PLATING PROCESSES, PAINT STRIPPERS AND OTHER AIRCRAFT MAINTENANCE CHEMICALS

by

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Approved for Public Release

* Materials Research Laboratories

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SUMMARY

The present work demonstrates how a slow-strain rate tensile test can be used to quantify rapidly in a statistically rigorous manner the severity of hydrogen embrittlement produced in high strength 4340 steel by paint strippers and plating processes. The results of multiple slow strain rate tests conducted at a crosshead displacement rate of 2×10^{-5} mm/s using notched specimens in various paint strippers show that a minimum mean fracture stress and maximum standard deviation can be defined that correlates with the pass/fail criterion in standard notched C-ring tests. The advantages of using a slow strain rate test as a viable alternative to existing standard methods for hydrogen embrittlement testing are discussed.

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1. INTRODUCTION

Recent trends in alloy development in the aircraft industry have been directed towards the production of alloys with high strength-to-weight ratios. Although many high strength low-alloy steels have been developed for use in aircraft structure, a lack of proper consideration regarding the detrimental effect of environment on their load-bearing properties has sometimes been evident. In particular, absorption of small amounts of hydrogen (few parts per million) can cause severe embrittlement in steels, particularly if the strength level exceeds 1200 MN/m^2. Hydrogen can be introduced into steel components by pickling and plating treatments, exposure to paint strippers and other maintainance chemicals and by corrosion during service. It is therefore important to minimise the amount of hydrogen introduced into the steel by such processes. Consequently, a variety of standard mechanical tests have been developed in the USA to determine the degree of embrittlement produced by plating processes and by fluids that come in contact with the steel [1-19]. Although test methods are continually updated and modified to accommodate the latest developments in engineering practice, it is often difficult to choose the most applicable specification for a particular circumstance. Closer examination reveals that this is due to the many differences in test method outlined in the various specifications (Tables 1 and 2):

a) The user must choose one of four specimen types: (i) notched tensile, (ii) notched bend, (iii) notched C-ring and (iv) smooth O-ring. The majority of specifications favour the notched tensile specimen (Tables 1 and 2).

b) Some specifications require specimens to be manufactured from 4340 steel heat treated to a tensile strength of 1800-1930 MN/m^2 whereas others specify use of any high strength steel. It is known that 4340 steel in this heat-treated condition is particularly susceptible to hydrogen embrittlement, hence standard tests using this steel should provide a sensitive test for determining the severity of embrittlement produced by plating processes, paint strippers and other aircraft maintenance chemicals.

c) When notched tensile specimens are used to assess the degree of embrittlement produced by plating processes, the number of specimens to be tested varies from a minimum of 2 to a maximum of 16 (Table 1). In all these tests, specimens are loaded to 75% of the notch tensile strength and usually held for 200h with fracture of one specimen being deemed sufficient to cause rejection of the particular plating and baking treatment.

d) A survey of specifications designed to assess the degree of hydrogen embrittlement produced by paint strippers shows that the method of applying the paint stripper, the applied stress and test time all vary with each specification. A summary of test procedures for each specification listed in Table 2 is given in the Appendix.

In all the specifications outlined above, tests produce a pass/fail result for the particular environmental condition under study. If all specimens fail, then the time to failure can provide some indication of the degree of embrittlement; however, if all specimens pass the test, there is no method of determining which environment induces the least amount of embrittlement. Consequently, little can be done to quantify the degree of embrittlement more precisely or treat the results in a statistically rigorous manner. Furthermore, since some tests can take up to 200h to perform, these methods cannot be used for assessing plating baths or maintainance ci.. nicals on a regular daily or weekly basis.

It is apparent from the above discussion that existing American specifications define test methods that display significant differences between the various specifications, can be time consuming and fail to provide a reliable estimate of the degree of embrittlement produced by plating procedures and embrittling fluids. Therefore, there is some justification for seeking an alternative mode of testing that will overcome the problems associated with the existing standard test methods.

Internal hydrogen embrittlement is known to involve a time-dependent diffusion process [20] and, hence, the rate of loading a specimen should provide a simple method for controlling the degree of severity of the test [21]. For steels not containing hydrogen, the rate of loading in a standard tensile test does not usually affect the tensile properties of the steel. For specimens containing hydrogen in which the tensile test is conducted sufficiently slowly, the hydrogen has time to diffuse to areas where the steel can become embrittled (usually grain boundaries) resulting in premature failure. If the loading rate is increased, the hydrogen has less time to damage the steel and the unembrittled properties of the steel are retained. This approach has been used in the French Standard Specification AIR 0825[12] to assess the hydrogen embrittlement susceptibility of high-strength steels to various electrolytic plating treatments. In this case, one end of a rectangular bar is clamped while the other end is loaded in bending at deflection rates ranging from 0.1 - 0.3 degree/s. The degree of embrittlement is obtained by comparing the angles at which fracture occurs for the embrittled and unembrittled specimens respectively.

The present work endeavours to bridge the gap between the French Specification AIR 0825 and the American specifications for hydrogen embrittlement testing. This is accomplished by applying the slow strain rate method to both notched and un-notched 4340 steel tensile specimens to see if a more precise assessment of the degree of embrittlement can be determined and to compare these results with those obtained using notched C-rings that are tested in accordance with standard test methods.

2. EXPERIMENTAL

The 9.5mm round 4340 steel bar used in the present work complied with the MIL-S-5000 specification[22] and had a composition (wt. %) of 0.43C, 0.85Cr, 0.84Mn, 0.25Mo, 1.78Ni, <0.01P, 0.29Si, 0.009S, < 0.05Al, remainder Fe. Notched tensile specimens were manufactured to comply with the reduced load requirements for the Type Ia specimen detailed in the standard ASTM F-519-77 specification (Figure 1). This was accomplished by maintaining the notch concentration factor within the range 2.9-3.3. The notch was prepared by low-stress crush grinding after heat-treatment. This process was carried out using an A60IV wheel rotated at 13-15 m/s and a jet of high sulphurised oil (Shell Garia 902) which was passed over the workpiece at a rate of 0.23 l/s to maintain the workpiece temperature below 150°C. The workpiece speed was maintained within the range 0.36-0.51 m/s. The last 0.25mm of stock was removed from the surface as follows: 0.20mm was removed at a rate of 0.012mm/pass and the remaining 0.05mm was removed at a rate of 0.005mm/pass. A grinding fluid entrainment baffle was fitted astride the grinding wheel to create a vacuum that sucked the grinding fluid into the wheel-work interface. Un-notched tensile specimens were manufactured with a specimen length of 100 mm and a central gauge length of 12.7mm (Figure 1). The diameter of the central section was initially machined to 3.4mm diameter prior to heat treatment. Low-stress crush grinding was used to reduce the diameter across the gauge length to 3.2mm after heat-treatment. Specimens were then polished with 600 grit silicon carbide paper and the minimum diameter across the gauge length measured prior to testing. Specimen heat treatment involved austenitisation at 815°C/1h in salt, oil quenching to 40-60°C followed by a double temper at 260°C/1+1h with air cooling to 20°C between tempers.

Specimens were preloaded to 0.5kN prior to testing to failure in a 20kN motor-driven hard-beam tensile testing machine which had a variety of gears enabling tests to be performed over a range of crosshead displacement rates varying from 10^{-2} to 10^{-6} mm/s. Loads were measured directly using a 22kN capacity load cell. The degree of embrittlement was quantified by the drop in fracture stress in notched tensile specimens and the drop in reduction in area at fracture in un-notched specimens when compared with equivalent values obtained with unembrittled specimens tested in air. With both types of specimen, a measure of the degree of embrittlement could also be obtained by measuring the percentage area of intergranular fracture on scanning electron micrographs.

2.1 Plating Treatments

The following plating, baking and passivation treatments were investigated to quantify the degree of hydrogen embrittlement produced in 4340 steel.

2.1.1 Low embrittlement cadmium electroplating solutions: specimens were cadmium plated to a thickness of 12-15 μm in low-embrittlement cadmium plating baths in accordance with Qantas Processing Specification P65[13] and Hawker de Havilland Process Specification HPS 1.03.00[14]. Both these specifications require baking at 190°C/23h within 4h of the plating treatment followed by chromate passivation[23]. The HPS 1.03.00 specification meets the hydrogen embrittlement requirements of the U.S. specifications MIL-STD-870A[15] and DPS 9.28[16] whereas the Qantas P65 specification meets the intent of the specifications BAC 5718[18], DPS 9.28[16], QQ-P-416[7] and AMS 2401[19].

2.1.2 Cadmium electroplating solutions with added brighteners: the addition of brighteners to cadmium electroplating baths causes more hydrogen to be generated during deposition of the cadmium coating. Due to the low porosity of this coating, significant quantities of hydrogen can be retained in the steel after baking resulting in an increased susceptibility to hydrogen embrittlement.[24] Specimens were plated in accordance with the UK Ministry of Aviation Specification DTD 904C[25]. Slow strain rate tests were also conducted to assess the effect of bakeout time on the degree of embrittlement.

2.1.3 Electroless nickel plating solutions: slow strain rate tests were performed to determine the degree of embrittlement induced in 4340 steel by the electroless nickel plating schedule outlined in the US Military Specification MIL-C-26074B[17]. In these tests, specimens were shot-peened, given a vapour degrease, an anodic alkaline clean and a nickel chloride strike before plating for 2h at 90°C in an acidic (pH 4-5) plating bath. The effects of chemically stripping the nickel plate, replating with nickel and baking at 190°C and 340°C were investigated.

2.2 Paint Strippers

Tests with paint strippers were only conducted using specimens given the low-embrittlement cadmium electroplating treatment. A 40 ml teflon chamber was used to expose the central 25mm portion of tensile specimens to the paint stripper. 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. A teflon lid also fitted over the top of the cell. Specimens were tested at several crosshead displacement rates in the presence of various proprietary paint stripper (A, B, C1, C2, D and E). Samples C1 and C2 were two different batches of the same paint stripper and had the same nominal composition. Paint strippers A and B had a phenolic base whereas the remainder were classified as non-phenolic paint strippers. The mean fracture stress and standard deviation were calculated from the results of testing three specimens in an identical manner. The severity of embrittlement produced in 4340 steel by paint strippers was also assessed according to a modified version of the K-55 specification (see Appendix) using tnree notched C-ring specimens.

3. RESULTS

3.1 Notched Specimens

3.1.1 Bare Steel Specimens Tested in Air

The results of testing notched bare steel specimens in air showed that the fracture stress was independent of crosshead displacement rate over the range 2×10^{-3} to 2×10^{-6} mm/s. The results of 21 specimens taken from several specimen batches heat treated at different times produced a mean value of 2456 MN/m^2 with a standard deviation of 52 MN/m^2 (Figure 2). The fracture mode of broken bare steel specimens was found to be 100% transgranular microvoid coalescence.

3.1.2 Plating Treatments

Slow strain rate tests conducted in air using specimens given the low-embrittlement cadmium plating treatment followed by baking at 190°C/23h produced similar fracture stress values to bare steel specimens tested in air at all crosshead displacement rates except the lowest rate used (2×10^{-6}mm/s) where the fracture stress was 25% lower. This result shows that not all the hydrogen introduced into the steel during the plating treatment was removed by baking. Similar tests conducted at a crosshead displacement rate of 2×10^{-6}mm/s with chromate-passivated specimens produced a greater drop in fracture stress compared with non-passivated specimens (Figure 2).

Slow strain rate tests conducted with specimens cadmium plated in a bath containing brighteners and subsequently baked at 190°C/23h produced low values for the fracture stress when tested in air at a crosshead displacement rate of 2×10^{-4} mm/s (Figure 2). Although an increase in the baking time to 100h increased the fracture stress of specimens subsequently tested in air at 2×10^{-4}mm/s, a comparison of fracture stress values of tests conducted at lower crosshead displacement rates indicated that this treatment was significantly more embrittling than the porous cadmium plating and passivation procedures.

Slow strain rate tests conducted in air at a crosshead displacement rate of 2×10^{-4} mm/s with a specimen subjected to the electroless nickel plating procedure resulted in a reduction in fracture stress of 40% compared with similar tests with bare steel specimens (Figure 3). Furthermore, the degree of embrittlement was increased when the nickel plate was stripped and the specimen replated. Although baking at 190^{o}C/3h increased the fracture stress to 88% of the unembrittled value, identification of a small area of intergranular fracture confirmed that sufficient hydrogen was present to initiate premature failure (Figure 4). Further baking at 340^{o}C/1h increased the fracture stress to 2330 MN/m^2. Since no positive identifications of intergranular fracture could be found in this case, it is postulated that the degree of hydrogen embrittlement was minimal and that the steel had been softened by tempering.

3.1.3 Paint Strippers

Results of notched C-ring tests with the six paint strippers indicated that paint strippers A,C1,D and E could be classified as 'non-embrittling' whereas paint strippers B and C2 could be labelled 'embrittling' (Table 3).

The effect of crosshead displacement rate on the mean fracture stress of cadmium-plated-and-baked notched tensile specimens in paint strippers A and B are shown in Figure 5. Although both paint strippers showed no signs of embrittling the steel when specimens were tested at 2×10^{-3} mm/s, tests at lower crosshead displacement rates in paint stripper B produced lower mean fracture stresses than in equivalent tests using paint stripper A. The maximum separation in mean fracture stress between the two paint strippers was observed at a crosshead displacement rate of 2×10^{-4} mm/s and hence this rate of straining was chosen for all subsequent tests using notched specimens.

The effect of notch radius on mean fracture stress was studied for unplated specimens tested in air and for cadmium plated-and-baked specimens tested in paint stripper A (Figure 6). Results showed that a 100% increase in notch radius produced only an 8% increase in mean fracture stress of bare steel specimens tested in air, whereas the mean fracture stress of cadmium plated-and-baked specimens tested in paint stripper A increased from 1600 to 2120 MN/m^2. Within the limitations of specimen design outlined in the ASTM F-519-77 specification (notch stress concentration factor within the range 2.9-3.3), the mean fracture stress for cadmium plated-and-baked specimens tested in paint stripper A is contained within a much smaller range (1872-2026 MN/m^2). The most significant decrease in mean fracture stress occurred when the notch concentration factor exceeded 3.3. Since the notch radius can be machined reproducibly within ±0.01mm, there is little problem in preparing specimens within the limitations laid down in the ASTM F-519-77 specification.

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 to determine the critical mean fracture stress that corresponded to the pass/fail criterion in the notched C-ring test. Results indicated that this value of the mean fracture stress lay in the range 1700-1850 MN/m^2 (Figure 7). Evaluation of the standard deviation for all paint stripper tests showed that the standard deviation was low for non-embrittling and highly-embrittling paint strippers whereas moderately-embrittling paint strippers produced results with high scatter (Figure 8).

Further slow strain rate experiments were conducted with embrittling paint stripper B to determine the cause of embrittlement. Tests conducted with bare steel notched specimens at a crosshead displacement rate of 2×10^{-4} mm/s in paint stripper B showed no evidence of embrittlement (Figure 9). When a steel specimen was galvanically coupled to a piece of cadmium of equivalent area during straining, premature failure of the steel specimen resulted. Finally, exposure of a cadmium plated-and-baked specimen to paint stripper B for a week resulted in premature failure after the specimen was cleaned, dried and subsequently tested in air. Similar results were also obtained using paint stripper A. These results, in addition to previous work involving pH/potential measurements in paint strippers[26], confirm that hydrogen is generated at the steel surface due to galvanic coupling between the steel and the cadmium in the paint stripper. The large anodic area of the cadmium provides the driving force for high cathodic current densities to be maintained at the small areas of steel exposed to the paint stripper ultimately leading to embrittlement of the steel.

3.2 Un-notched Specimens

A limited number of tests were conducted using un-notched specimens for comparison with the results of notched specimens. Slow strain rate tests using un-notched bare steel specimens and porous cadmium plated-and-baked specimens tested in air at crosshead displacement rates between 2×10^{-5} mm/s and 2×10^{-3} mm/s (1.6×10^{-6} and 1.6×10^{-4} strain rate) produced cup-cone type fractures with reduction in area at fracture varying between 42 and 50% (Figure 10). Since no sign of intergranular fracture was evident, these reduction in area values were used as a basis for determining the degree of embrittlement of plated specimens tested in paint strippers. When both bare steel specimens and cadmium plated-and-baked specimens were tested at a crosshead displacement rate of 2×10^{-6} mm/s (1.6×10^{-7} strain rate), specimens failed prematurely to give a shear-like fracture across the full width of the specimen. Since no traces of intergranular fracture were observed in the bare steel specimens, embrittlement by hydrogen was not considered to be responsible for this type of fracture. In the case of the cadmium plated-and-baked specimens tested at 2×10^{-6} mm/s, small areas of intergranular crack initiation were observed prior to the onset of fast shear-like fracture across the remainder of the specimen.

The effect of crosshead displacement rate on reduction in area was determined for specimens tested in paint strippers A and B (Figure 10). At a crosshead displacement rate of 2×10^{-3} mm/s, no decrease in the reduction in area at fracture was observed for cadmium plated-and-baked specimens tested in either paint stripper compared with unplated specimens tested in air. Tests with the more embrittling paint stripper B showed that the reduction in area decreased rapidly for crosshead displacement rates below 7×10^{-4} mm/s, whereas the corresponding rate for paint stripper A was $\sim 5 \times 10^{-5}$ mm/s. At lower crosshead displacement rates, the reduction in area values for paint stripper A approached those for the embrittling paint stripper B. The drop in reduction in area values could also be correlated with the proportion of intergranular fracture on the fracture surface of embrittled specimens with the amount varying from approximately 0.05% in mildly embrittled specimens to 10% in cases of extreme embrittlement.

4. DISCUSSION

The purpose of the present paper is to assess the potential of the slow strain rate technique for quantifying the degree of hydrogen embrittlement in ultra-high strength 4340 steel and then to compare the merits of this technique with methods already being used in existing specifications. A comparison of results for slow strain rate testing of notched and un-notched specimens highlights a number of factors that favour the notched specimen as a more suitable choice for assessing the degree of embrittlement:

a) The fracture stress determined in tests with notched specimens is a relatively simple parameter to measure compared with the more complicated procedure to determine the reduction in area at fracture using un-notched specimens.

b) The fracture stress of unembrittled notched specimens is reproducible to ±2% compared with ±10% for the reduction in area at fracture associated with un-notched specimens.

c) Anomalous shear-type fractures occur at low strain rates with unembrittled un-notched specimens making assessment of the degree of embrittlement dubious.

d) Tests conducted with paint stripper A show that embrittlement only becomes evident for un-notched specimens at crosshead displacement rates $< 5\times10^{-5}$ mm/s compared with 7×10^{-4} mm/s for notched specimens. Consequently, assessment of the degree of embrittlement with un-notched specimens in paint strippers could take up to 20 times longer than equivalent tests with notched specimens. At a crosshead displacement rate of 2×10^{-4} mm/s, a maximum time of only 2.5h is required to test a notched specimen.

The scatter in results for both notched and un-notched specimens tested in paint strippers varies with applied strain rate and can become unacceptably high in certain circumstances. Results obtained with un-notched specimens tested in paint stripper B show a narrow region ($3\text{-}6\times10^{-4}$ mm/s) where the reduction in area at fracture becomes very sensitive to small changes in crosshead displacement rate (Figure 10). Consequently, if tests were conducted at a constant crosshead displacement rate within this narrow band, the reduction in area at fracture would be very sensitive to small changes in hydrogen concentration in the steel. The same argument would also apply to un-notched specimens tested in paint stripper A at a crosshead displacement rate of 2×10^{-5} mm/s and to notched specimens tested in paint stripper mixtures containing 65% C1 + 35% C2 and 75% C1 + 25% C2 at a crosshead displacement rate of 2×10^{-4} mm/s. This particular aspect could be used to advantage in any standard test for assessing susceptibility to hydrogen embrittlement using notched specimens. In addition to defining a minimum fracture stress for acceptance of a given paint stripper, results displaying a high standard deviation would provide additional evidence that a particular plating treatment or paint stripper may be unacceptable. Consequently, for slow strain rate tests conducted at a crosshead displacement rate of 2×10^{-4} mm/s, where failure in the range 1700-1850 MN/m^2 corresponds to the pass/fail criterion in the notched C-ring test, it is suggested that a maximum standard deviation of 300 MN/m^2 be specified as a possible additional criterion for acceptance. Due to the significance of defining a minimum mean fracture stress and a maximum standard deviation as possible criteria for acceptance or rejection of products causing hydrogen embrittlement, it is suggested that better statistical values might be obtained from the results of five rather than three specimens. This method of assessing scatter of results is considered superior to the method of assessment laid down on ASTM F-519-77 where retesting of a further batch of three specimens is required if one specimen fails during testing of the initial batch. Furthermore, this latter procedure can take up to two weeks to complete compared with 2.5h for a slow strain rate test performed at a crosshead displacement rate of 2×10^{-4} mm/s. Manufacturing costs for the notched tensile specimen have also been found to be 50% less than for the notched C-ring specimen. This is due to the lower cost of machining (40%) and lower rejection rate for the notched tensile specimen (<0.5%) compared with the notched C-ring specimen (5%). A further advantage of the slow strain rate method is the ability to compare quantitatively the degree of embrittlement produced by different plating treatments or paint strippers irrespective of the amount of hydrogen present in the steel. Although time to failure provides some indication of the degree of embrittlement in severely embrittled C-ring specimens, no comparison is possible for treatments that pass the standard tests.

The slow strain rate method could also be used to quantify the relative severity of other procedures for testing paint strippers laid down in ASTM F-519-77, MIL-R-83936B Amendment 1, MIL-R-25134B and MIL-R-81294B specifications. This could be done by either (a) conducting tests at a crosshead displacement rate of 2×10^{-4} mm/s and defining an appropriate minimum fracture stress and maximum standard deviation consistent with the pass/fail results of the standard specifications, or (b) altering the crosshead displacement rate and maintaining the current values for the minimum fracture stress and maximum standard deviation. Justification for this approach is evident from a comparison of calculated notch stress concentration factors in five notched tensile specimens detailed in the standard specifications (Table 4). Stress concentration factors for the five specimen geometries are calculated assuming the notched specimen to comply with Neuber's solution for a grooved shaft loaded in tension[27] and found to vary from 2.5 to 3.9 (Figure 11). Troiano[20] has demonstrated that time to failure of sharply-notched embrittled steel specimens loaded to the same percentage value of the unembrittled fracture stress decreases with increasing notch stress concentration factor. Consequently, it is unlikely that the same pass/fail result would occur if all five specimen types were used to assess treatments producing the same degree of embrittlement.

A comparison of the time to failure of chromium-plated notched tensile and C-ring 4340 steel specimens also illustrates the difference in susceptibility to hydrogen embrittlement between these two types of specimen (Figure 12)[28]. Although both curves approach the same lower critical stress at long testing times, the shorter times to failure of notched C-ring specimens tested at the same applied stress demonstrates that this specimen type provides a more severe test compared with identical tests using notched tensile specimens. This result is surprising since the notch stress concentration factor in the tensile specimen is significantly higher than in the C-ring specimen. Consequently, it seems possible that notched tensile specimens subjected to a less embrittling treatment and tested in accordance with the ASTM F-519-77 specification could pass the standard test whereas notched C-ring specimens would fail the same test. At the present time, it is apparent that the degree of test severity associated with the various specimens and testing methods is a quantity that has been neither properly defined nor adequately investigated.

The ability of the slow strain rate method to control the severity of testing by appropriate choice of crosshead displacement rate allows comparative studies of different embrittling treatments to be made. In the case of cadmium plating treatments that are designed to induce minimal hydrogen embrittlement in ultra-high strength steels, standard specifications[6-8,13-16,18,19] stipulate the use of porous cadmium plating treatments followed by baking at 190°C/23h. Slow strain rate tests with notched 4340 steel specimens given this treatment show no signs of embrittlement when tested at crosshead displacement rates ~2×10^{-5} mm/s. The 25% reduction in fracture stress observed in tests conducted at 2×10^{-6} mm/s show that small quantities of hydrogen are retained in the specimen, but due to the extreme severity of this test, this condition is not considered detrimental to the integrity of components given this treatment. The lower fracture stresses obtained with cadmium plated-and-baked specimens that are also chromate passivated signifies that the latter process introduces further hydrogen into the steel. It is surprising that the degree of embrittlement is not greater since the chromate passivation bath is highly acidic (pH ~1). Since no reduction in fracture stress is observed in tests performed at a crosshead displacement rate of 2×10^{-4} mm/s, the degree of embrittlement is not considered to be as great as that produced by electroless nickel plating, bright cadmium plating-and-baking treatments or by paint strippers A,B,C1 and C2.

In contrast, bright cadmium plating followed by baking at 190°C/23h and electroless nickel plating without baking are not recommended for application on ultra-high strength steels, since excessive levels of hydrogen are introduced into the steel during the plating process. It is not surprising that fracture stress values < 1500 MN/m^2 are recorded for all notched tensile specimens tested at a crosshead rate of 2×10^{-4} mm/s. These treatments would be expected to fail standard notched C-ring tests bearing in mind that the test severity for assessment of plating treatments is greater than for aircraft maintainance chemicals and paint strippers (Tables 1 and 2). Slow strain rate tests also show that the degree of embrittlement in 4340 steel is significantly enhanced when electroless nickel coatings are stripped and specimens replated and also that baking at 190°C/3h is insufficient to remove all traces of hydrogen.

Bright cadmium plated 4340 steel specimens that are baked at $190^{o}C/100h$ display minimal reduction in fracture stress when tested at a crosshead displacement rate of 2×10^{-4} mm/s. The 40% reduction in fracture stress observed in tests conducted at 2×10^{-5} mm/s suggest that significant quantities of hydrogen are still present in the steel and hence this condition must be considered as a possible threat to the integrity of structure subjected to large loads for long periods of time (e.g. aircraft undercarriage structure or any high strength steel component sustaining residual tensile stresses). A number of cases have been recorded where notched C-rings, which passed the standard tests, were found to fail under load after storage for many weeks.[29] It is therefore important to know whether existing standard methods can adequately predict the long-term (months/years) durability of structure that is susceptible to hydrogen embrittlement. Future work should seek to correlate the fracture stress in a slow strain rate test with failure time in a standard hydrogen embrittlement test in which the test time is extended to many months. Since hydrogen embrittlement testing conditions invoked in the various standard tests for assessing plating treatments are more stringent than those for testing aircraft maintainance chemicals and paint strippers (Tables 1 and 2), slow strain rate tests for assessment of plating treatments should be carried out at a crosshead displacement rate $<2\times10^{-4}$ mm/s.

Before further comparisons of the slow strain rate method and conventional standard tests are made, consideration should be given to the location and rate of hydrogen generation during a test. Three situations are envisaged:

(a) finite initial distribution of hydrogen within the specimen e.g. as-plated and plated-and-baked specimen,

(b) galvanic generation of hydrogen at all plated surfaces exposed to an external environment e.g. exposure of cadmium-plated steel specimens to embrittling paint strippers, and

(c) generation of hydrogen in the notch region only e.g. exposure of aqueous Environments (e.g. distilled water) that cause stress corrosion cracking.

Since the source and spatial location of hydrogen is different in each of the three cases, the kinetics of hydrogen accumulation in the region of the notch would also be expected to vary. It is therefore possible that the critical slow strain rate fracture stress corresponding to the pass/fail condition in the standard constant load test may vary with the mode of hydrogen generation or distribution of hydrogen within the specimen. The present work has successfully determined a correlation between the standard notched C-ring test and the slow strain rate test for case (b), and it is recommended that future work should be aimed at determining a similar correlation for cases (a) and (c). It is anticipated that a different correlation between the two test methods might be expected in case (c) where generation of hydrogen depends on the interaction of the environment with fresh metal surface in the notch. Breakdown of a passivating film is more likely to occur in a slow strain rate test than a constant load or constant displacement test and hence the slow strain rate method is likely to generate less scatter and provide a more positive assessment of the degree of embrittlement.

5. CONCLUSIONS

A comparison of the slow strain rate method with standard constant load tests highlights many advantages of the former test in assessing susceptibility of ultra-high strength steels to hydrogen embrittlement:

(a) slow strain rate tests can be done using a notched tensile specimen that complies with the reduced load requirements in the ASTM F-519-77 specification,

(b) the slow strain rate test with notched specimens utilises the fracture stress as a simple parameter for assessing the degree of embrittlement,

(c) the severity of the slow strain rate test can be controlled by altering the crosshead displacement rate,

(d) for multiple testing of specimens at a given crosshead displacement rate, a minimum mean fracture stress and maximum standard deviation can be specified for acceptance of a paint stripper or plating process,

(e) quantitative results can be obtained in less than 3h with a slow strain rate test conducted at a crosshead displacement rate of 2×10^{-4}mm/s, compared with a maximum time of 100-300h for tests defined in the standard specifications,

(f) the slow strain rate technique could be used to quantify the relative severity of test procedures defined in the various standard specifications,

(g) an overall manufacturing cost reduction of 50% can be achieved with the use of notched tensile specimens compared with notched C-ring specimens.

It is argued that the evidence cited in this paper strongly supports the view that the slow strain rate method should be adopted as an alternative standard method for assessing the severity of hydrogen embrittlement produced by plating processes, paint strippers and other aircraft maintainance chemicals. It is hoped that these advances will lead to better qualification of products and processes used in the improved maintainance of aircraft components, thereby reducing the risk of failure by hydrogen embrittlement during subsequent service.

ACKNOWLEDGEMENTS

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APPENDIX

Details of U.S. Specifications for Determining the Degree of Hydrogen Embrittlement Induced by Paint Strippers and Aircraft Maintenance Chemicals in High Strength Steels

1. ASTM F-519-77[6]

Specification allows notched tensile, notched bend, notched C-ring and smooth O-ring 4340 steel specimens to be used. Specimens are porous cadmium plated-and-baked and chromate passivated. A minimum of three specimens are loaded in air to the values shown in Table 2 and the notched region immersed in the paint stripper or fluid. If two or more specimens fail within 150h, the environment is considered embrittling. If one specimen fails, a further three specimens are tested and if any specimen fracture during retest, the environment is considered embrittling.

2. MIL-R-83936B Amendment 1[4]

Four notched tensile steel specimens are cadmium plated-and-baked as outlined in MIL STD 870A[15]. Specimens are then immersed in paint stripper for 4h, rinsed, dried and loaded in air to 75% of the notch tensile strength. Failure of one specimen within a period of 200h is sufficient to cause rejection of the paint stripper.

3. MIL-R-25134B[5]

Four unplated and four cadmium plated-and-baked notched tensile 4340 steel specimens are loaded in air to 75% of the notch tensile strength. The paint stripper is applied to the notched region of the specimen, wiped off carefully after 10 minutes, and the process repeated to give four full cycles per day at two hourly intervals until either 100h has elapsed or until failure. If the specimens are still intact after the 100h period, the load is maintained for a further 100h and if any specimens fail, the paint strippers is considered to be embrittling.

4. MIL-R-81294B[3]

Four cadmium plated-and-baked 4340 steel notched C-rings are loaded to 75% of the notched breaking strength in air. The C-ring specimens are immersed for 60s with the notched side down to a point where the ring is covered approximately 25 mm on either side of the notch. The rings are removed and allowed to hang notched side down in air for 100h. Failure or cracking of any specimen within this period causes rejection of the paint stripper.

5. Australian RAAF Engineering Specification K55[11]

Identical to MIL-R-81294B[3] except that either 4340 steel or D6ac steel can be used. Ultimate tensile strength of D6ac steel is specified to be within range 1800-1930 MN/m^2.

6. Modified RAAF Specification K55

Identical to K55 specification except that three notched C-rings are tested for 100h and if two or more specimens crack or fail within this period, the paint stripper is considered embrittling. If one specimen fails, a further three specimens are tested and the paint stripper is considered acceptable only if all three specimens pass the 100h test.

TABLE 1 – Summary of Some American and Australian Standard Specifications for Hydrogen Embrittlement Testing of High Strength Steels Subjected to Plating Processes.

Specification	Specimen Geometry	Material	Stress Concentration Factor	Type of Loading	Plating Process	Applied Stress (% NTS)	Maximum Test Time(h)	No. of Specimens
MIL-S-5002[1]	N.T.	N/S	3.9	C.L.	Inorganic Coatings/ Surface Treatments	75	200	16
MIL-STD-1501B[9]	N.T.	4340	3.9	C.L.	Cr	75	200	16
MIL-C-26074B[17]	N.T.	N/S	3.9	C.L.	Electroless Ni	$\geq 20^{*}$	200	4
BAC 5804[10]	N.T.	4340	3.1	C.L.	Cd-Ti	75	150	2
BAC 5718[18]	N.T.	4340	3.1	C.L.	Cd	75	200	2
AMS 2401C[19]	N.T.	N/S	3.6	C.L.	Cd	75	200	4
QQ-P-416C[7]	N.T.	N/S	3.9	C.L.	Cd	75	200	4
HPS 1.03.00[14]/1.04.05[8]	N.T.	4340	3.9	C.L.	Cd	75	200	4
MIL-STD-870A[15]	N.T.	4340	3.9	C.L.	Cd	75	200	16
ASTM F-519-77 (1a)[6]	N.T.	4340	3.1	C.L.	Cd	75	200	3
ASTM F-519-77 (1b)	N.T.	4340	2.8	C.D.	Cd	75	200	3
ASTM F-519-77	N.B.	4340	3.9	C.D.	Cd	75	200	3
ASTM F-519-77	N.C-ring	4340	4	C.D.	Cd	75	200	3
ASTM F-519-77	S.O-ring	4340		C.D.	Cd	92 (UTS)	200	3
DPS 9.28[16]	S.O-ring	N/S		C.D.	Cd	92 (UTS)	168	4

N.T. - notched tensile
N.B. - notched bend
NTS - notch tensile/ bend strength

N.C-ring - notched C-ring
S.O-ring - smooth O-ring
N/S - not specified

C.L. constant load
C.D. - constant displacement
UTS - ultimate tensile strength

* The reason for choosing a minimum applied stress of 20% NTS is not clear.

TABLE 2 - Summary of Some American and Australian Standard Specifications for Hydrogen Embrittlement Testing of High Strength Steels Subjected to Aircraft Maintainance Chemicals and Paint Strippers.

Specification	Specimen Geometry	Material	Stress Concentration Factor	Type of Loading	Environment	Applied Stress (% NTS)	Maximum Test Time(h)	No. of Specimens
MIL-R-83936B[4]	N.T.	N.S	3.9	C.L.	P.S.	75	200	4
MIL-R-25134B[5]	N.T.	4340	2.5	C.L.	P.S.	75	200	8
ASTM F-519-77[6]	N.T.	4340	3.1	C.L.	A.M.C., P.S.	45	150	3 (→ 6)
ASTM F-519-77	N.T.	4340	2.8	C.D.	A.M.C., P.S.	55	150	3 (→ 6)
ASTM F-519-77	N.C-ring	4340	4	C.D.	A.M.C., P.S.	65	150	3 (→ 6)
MIL-R-81294B[3]	N.C-ring	4340	4	C.D.	P.S.	75	100	4
RAAF K-55[11]	N.C-ring	4340	4	C.D.	P.S.	75	100	4
Present work (Modified RAAF K-55)	N.C-ring	4340	4	C.D.	P.S.	75	100	3 (→ 6)
ASTM F-519-77	S.O-ring	4340		C.D.	A.M.C.,P.S.	[illegible]	150	3 (→ 6)

N.T. - notched tensile
N.B. - notched bend
N/S - not specified
N.C-ring - notched C-ring
S.O-ring - smooth O-ring
U.T.S. - ultimate tensile strength
P.S. - paint stripper
A.M.S. - aircraft maintainance chemicals
N.T.S. - notch tensile/bend strength
C.L. - constant load
C.D. - constant displacement

TABLE 3 - Results of Notched C-ring Tests in Paint Strippers

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	

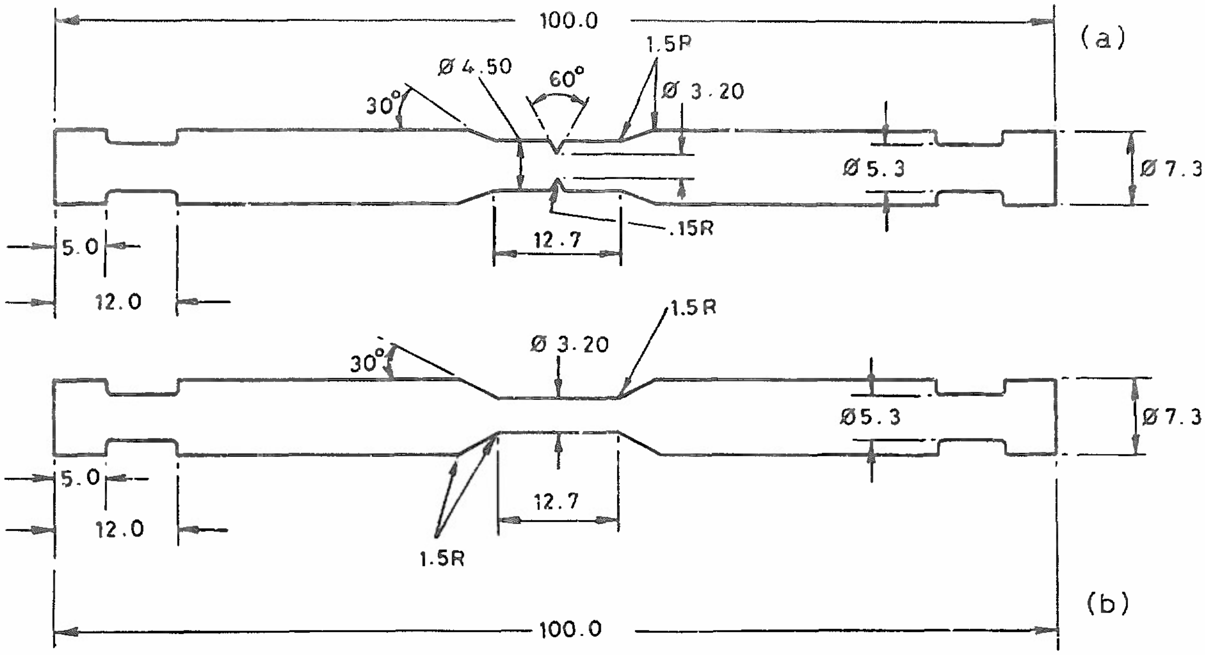
P : specimen unbroken after 100h

All specimens that passed the 100h test were also found to be intact after 150h.

TABLE 4 - Specimen Geometry and Stress Concentration Factor (k_T) of Notched Tensile Specimens Used in Standard Specifications for Hydrogen Embrittlement Testing of Ultra-High Strength Steels[27].

Specification	D(mm)	d(mm)	r(mm)	d/D	r/D	k_T
MIL-S-5002C) MIL-R-83936B) MIL-STD-870A) MIL-STD-1501B) MIL-C-26074B) QQ-P-416C) HPS 1.04.05)	12.50	8.84	0.25	0.707	0.020	3.9
AMS 2401C	12.7	9.07	0.30	0.714	0.024	3.6
BAC 5854) BAC 5804) ASTM F-519-77(Ia))	8.46	5.97	0.25	0.706	0.030	3.2
ASTM F-519-77(Ib)	4.78	2.27	0.15	0.476	0.032	2.8
MIL-R-25134B	9.07	5.72	0.64	0.631	0.071	2.5
Present Work	4.50	3.20	0.15	0.707	0.033	3.1

See Figure 12 for definition of D, d and r.



a) notched

b) un-notched

Figure 1. 4340 steel round tensile specimens:

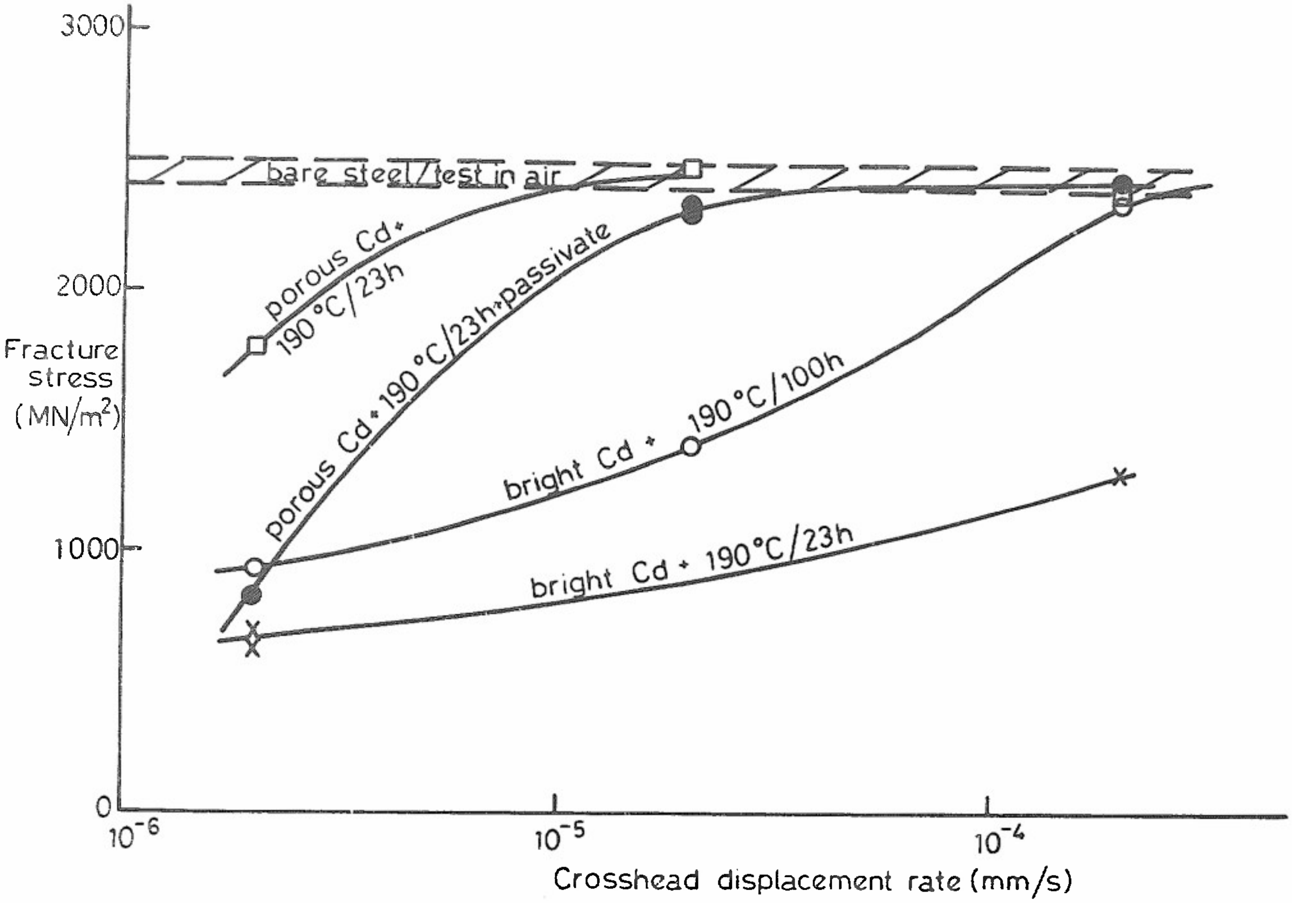


Figure 2. Fracture stress of cadmium plated-and-baked 4340 steel notched specimens tested in air at various crosshead displacement rates.

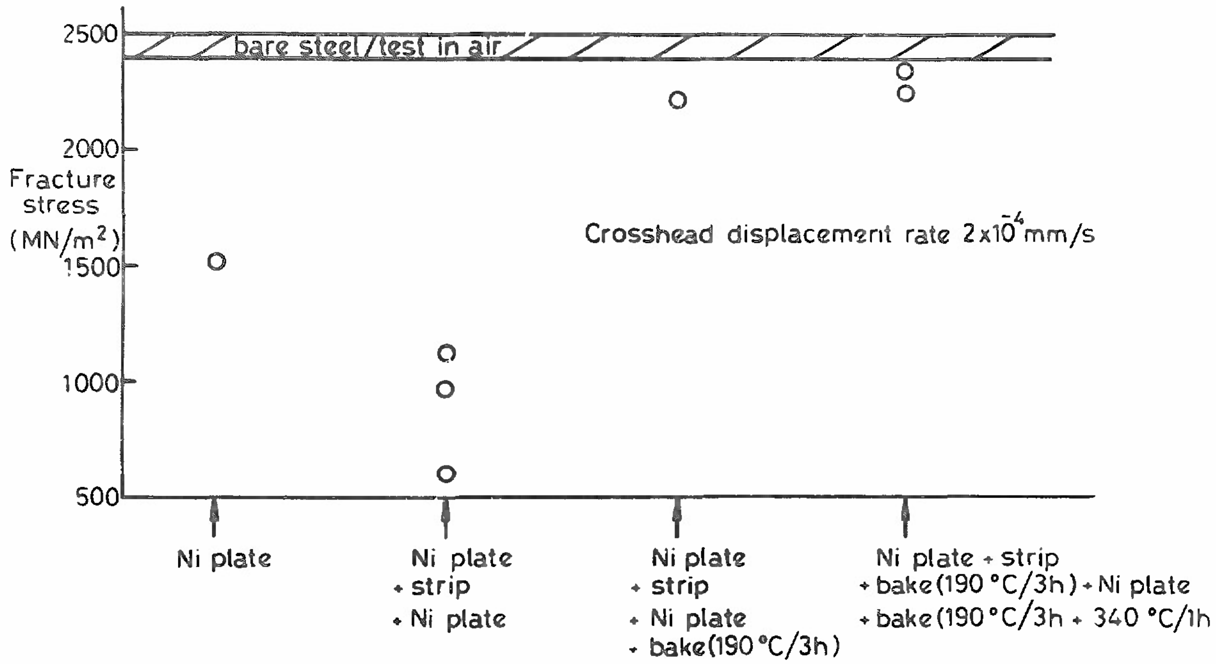


Figure 3. Fracture stress of electroless nickel plated 4340 steel notched specimens tested in air at a crosshead displacement rate of 2×10^{-4} mm/s.

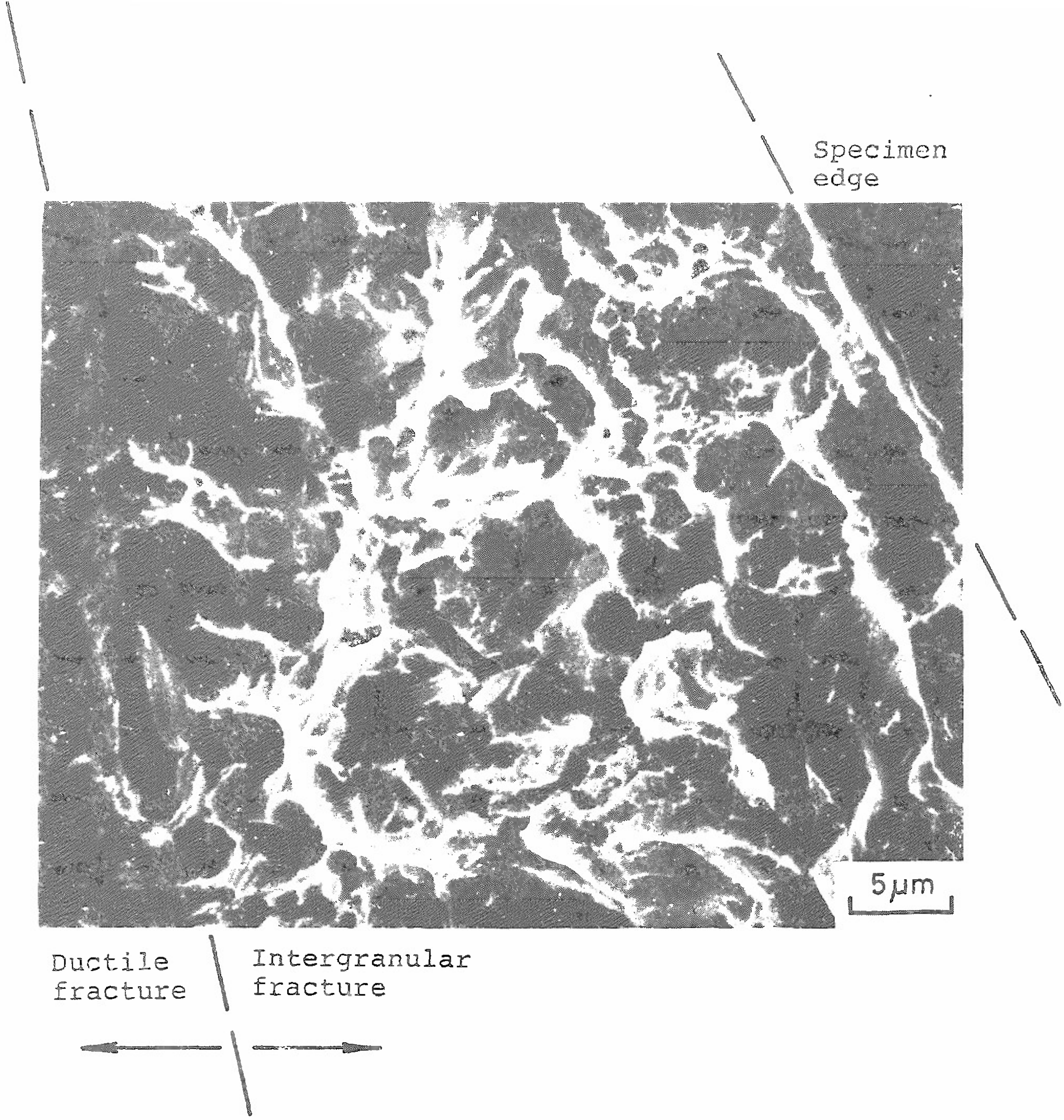


Figure 4. Intergranular fracture initiation site of 4340 steel notched specimen which had been nickel plated, stripped, replated, baked at 190°C/3h and tested in air at a crosshead displacement rate of $2x10^{-4}$ mm/s.

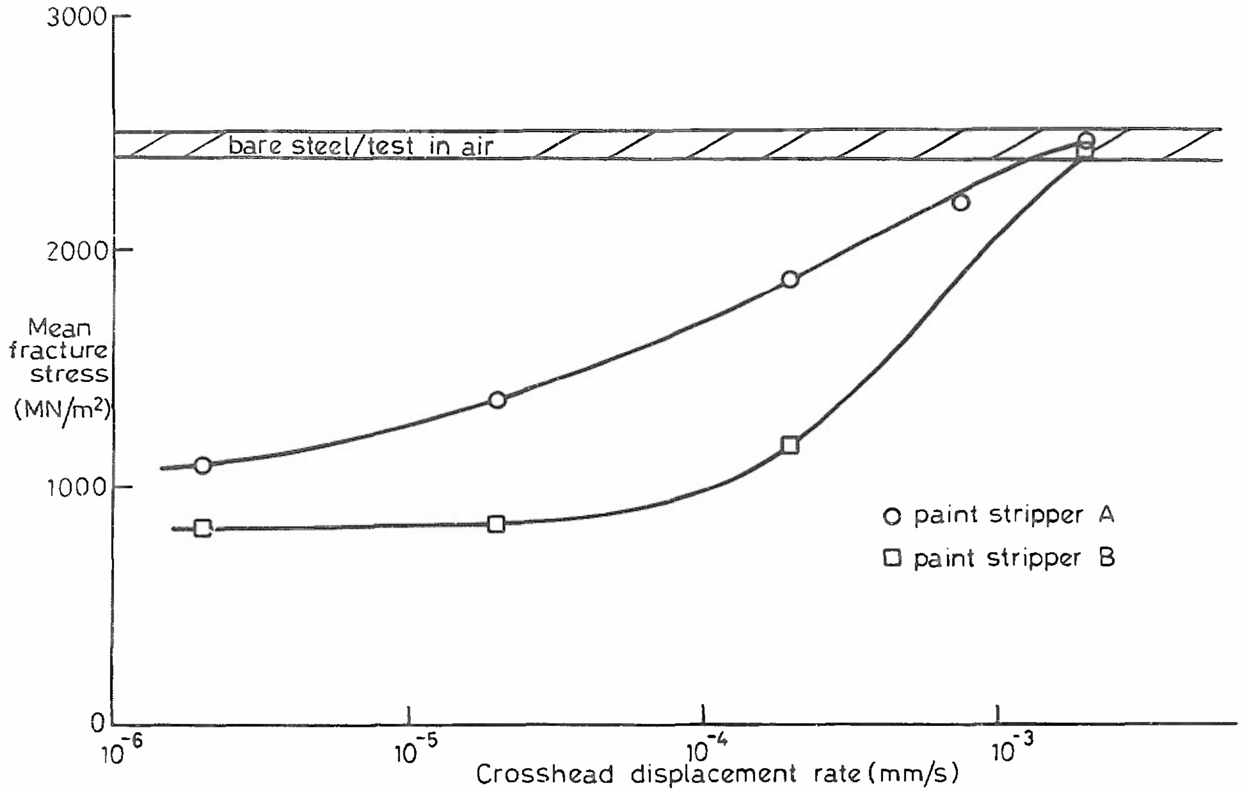


Figure 5. Mean fracture stress of cadmium plated-and-baked 4340 steel notched specimens tested in paint strippers A and B at various crosshead displacement rates.

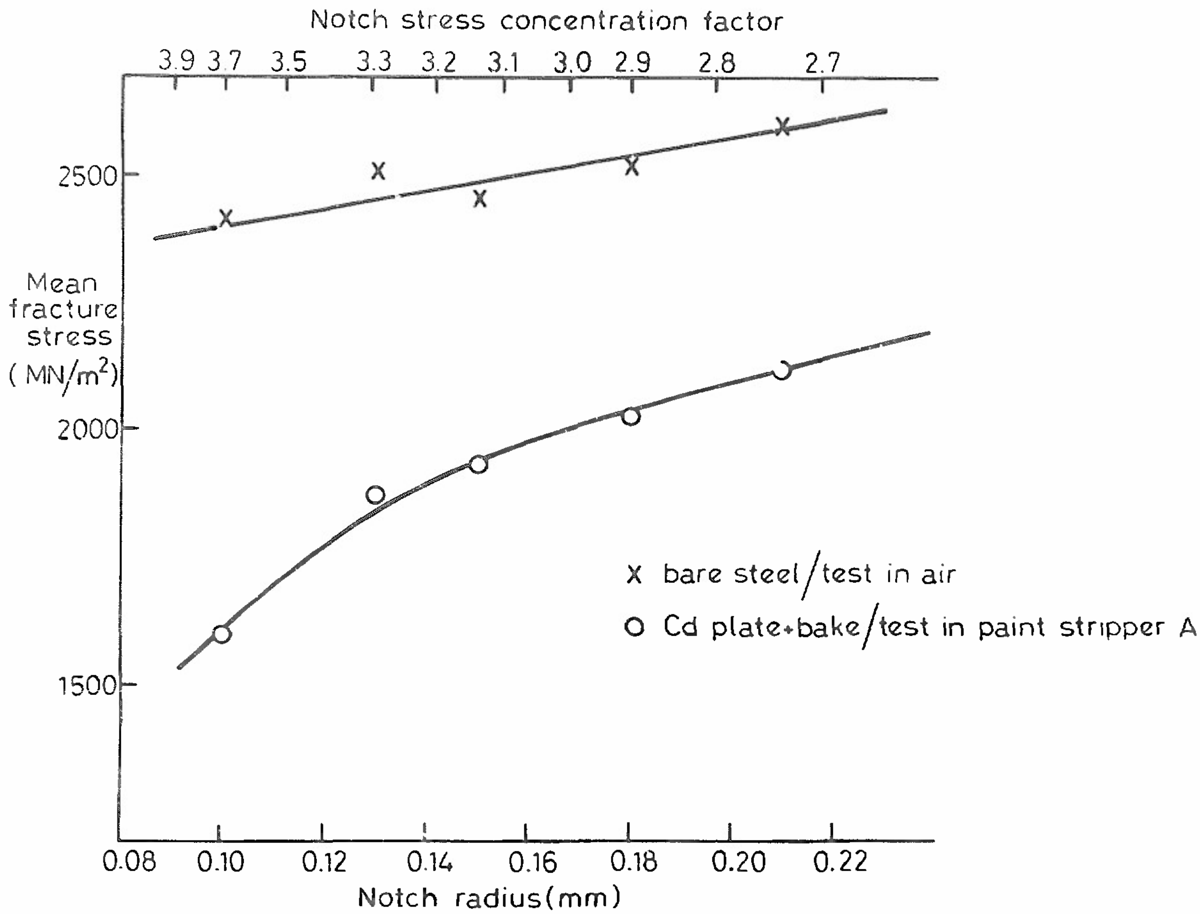


Figure 6. Effect of notch radius on mean fracture stress of 4340 bare steel specimens tested in air and cadmium plated-and-baked 4340 steel specimens tested in paint stripper A at a crosshead displacement rate of 2×10^{-4} mm/s.

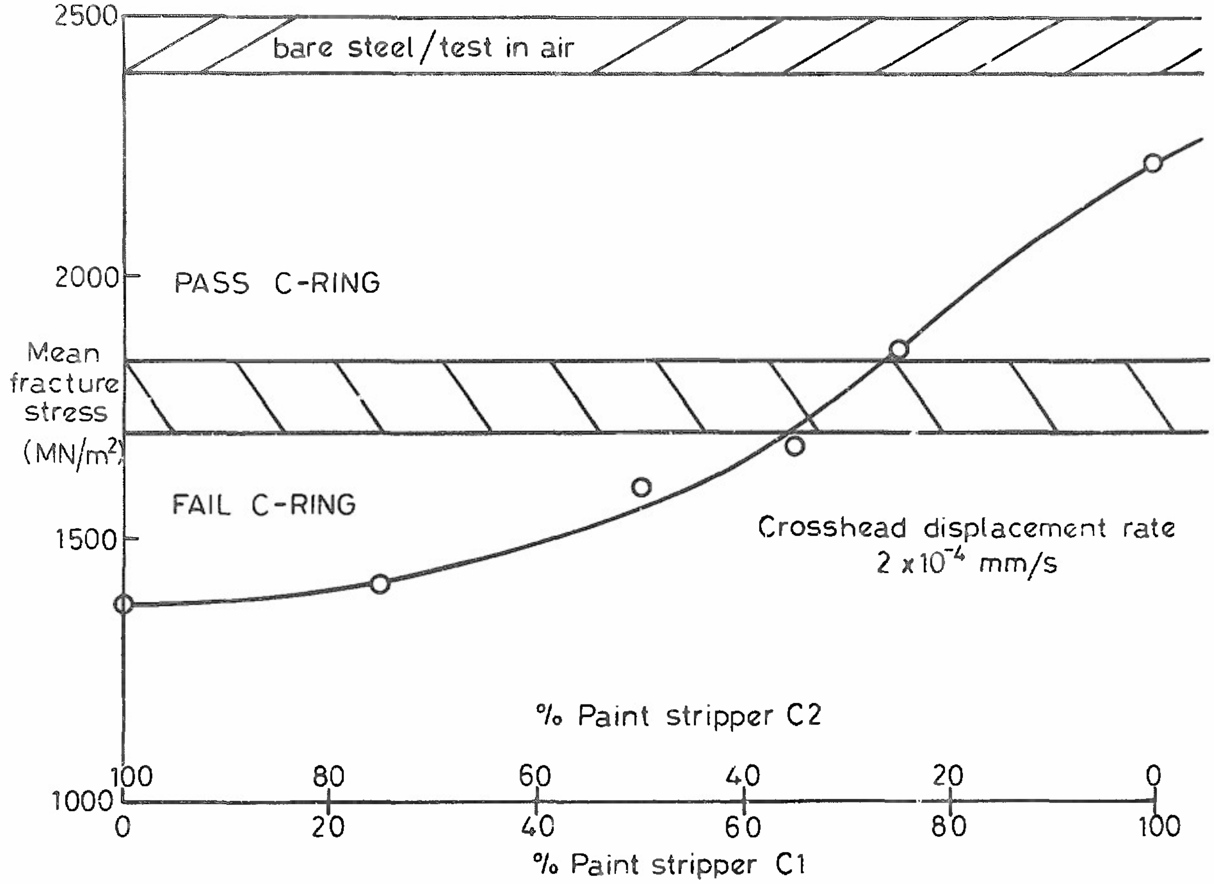


Figure 7. Comparison of notched C–ring and slow strain rate results in mixtures of paint strippers C1 and C2.

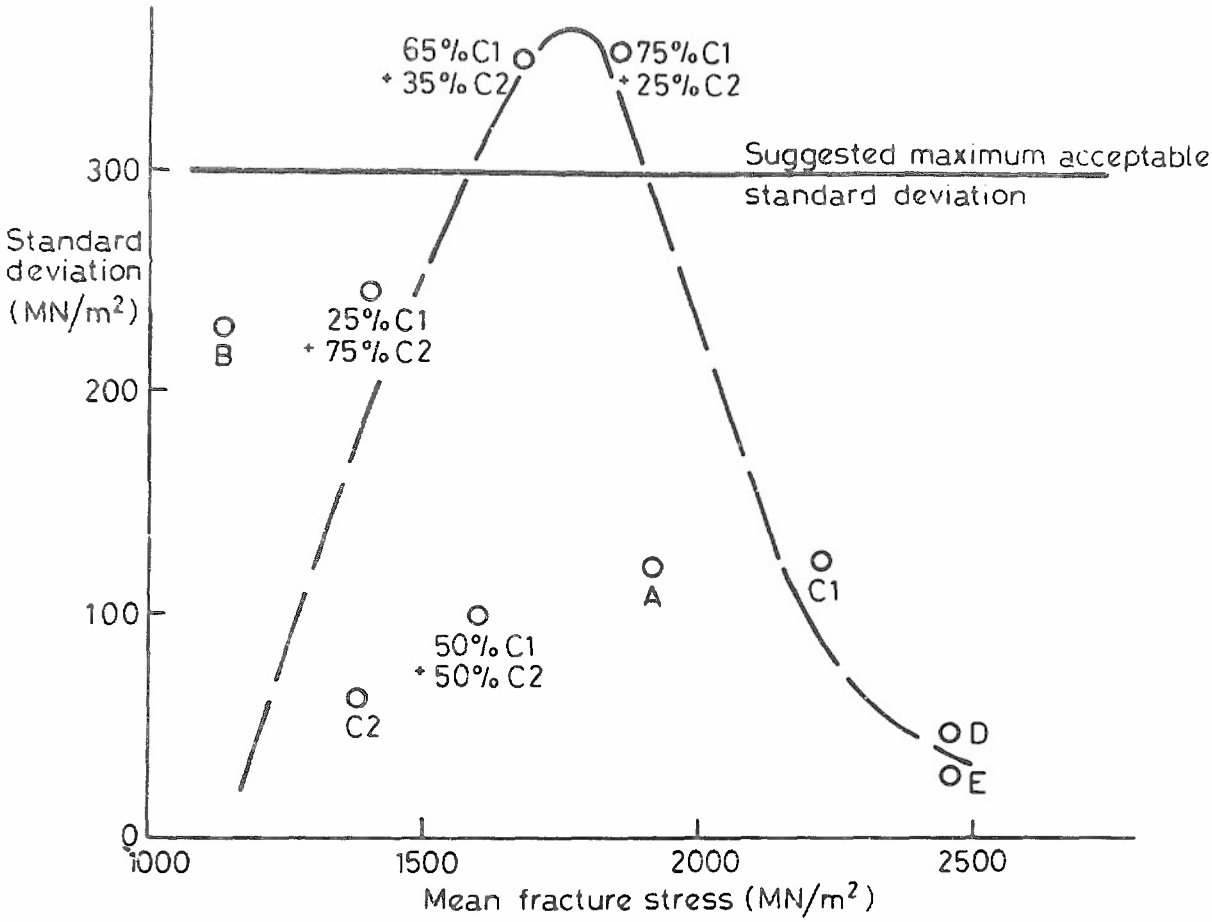


Figure 8. Mean fracture stress and standard deviation of cadmium plated-and-baked 4340 steel notched specimens tested in paint strippers at a crosshead displacement rate of 2×10^{-4} mm/s.

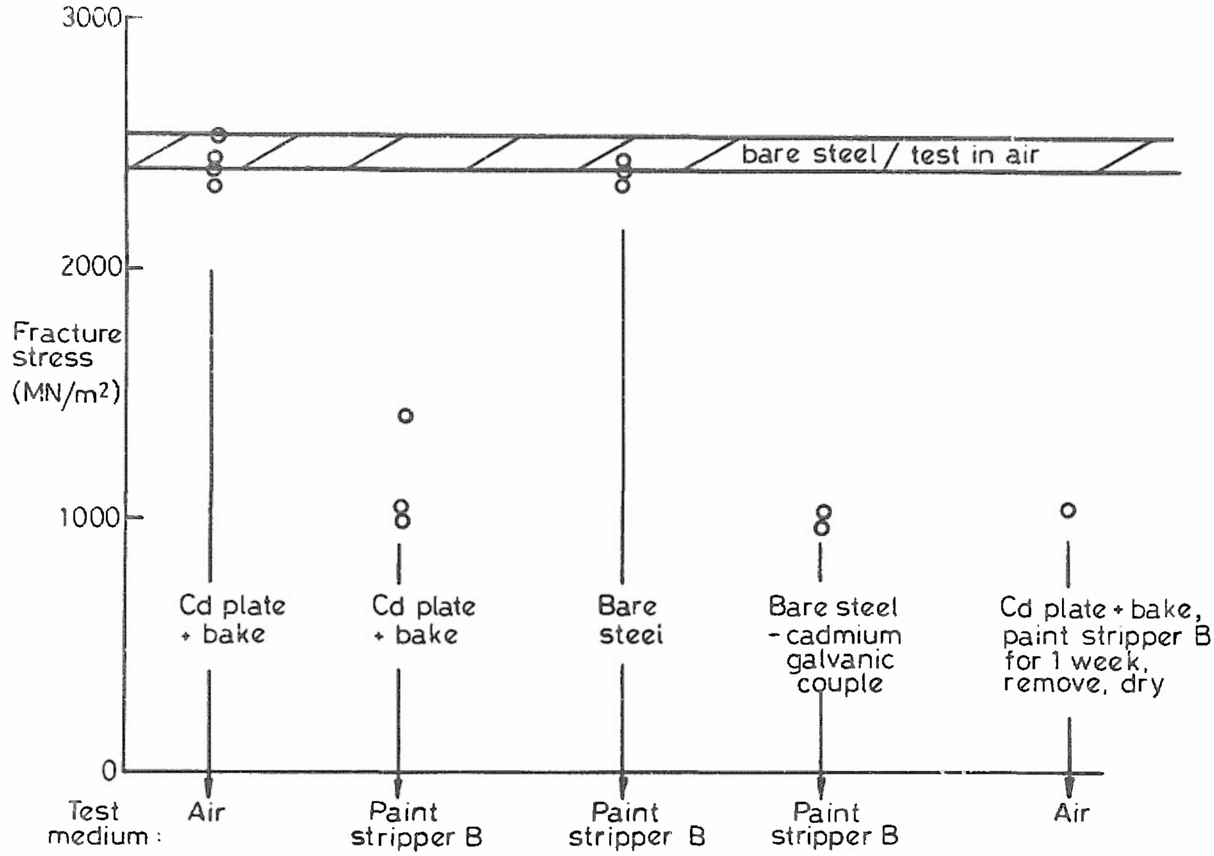


Figure 9. Fracture stress of 4340 steel notched specimens tested under various conditions at a crosshead displacement rate of $2x10^{-4}$ mm/s.

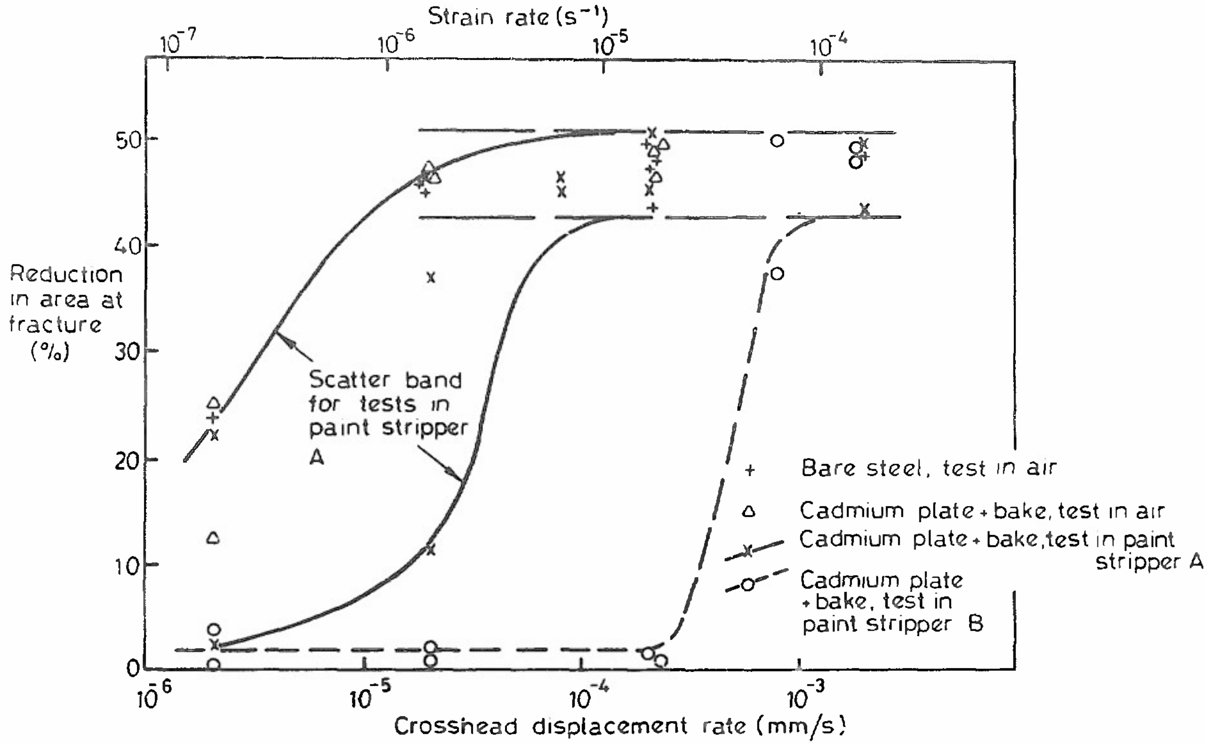


Figure 10. Percentage reduction in area at fracture of cadmium plated-and-baked 4340 steel unnotched specimens tested in paint strippers A and B at various crosshead displacement rates.

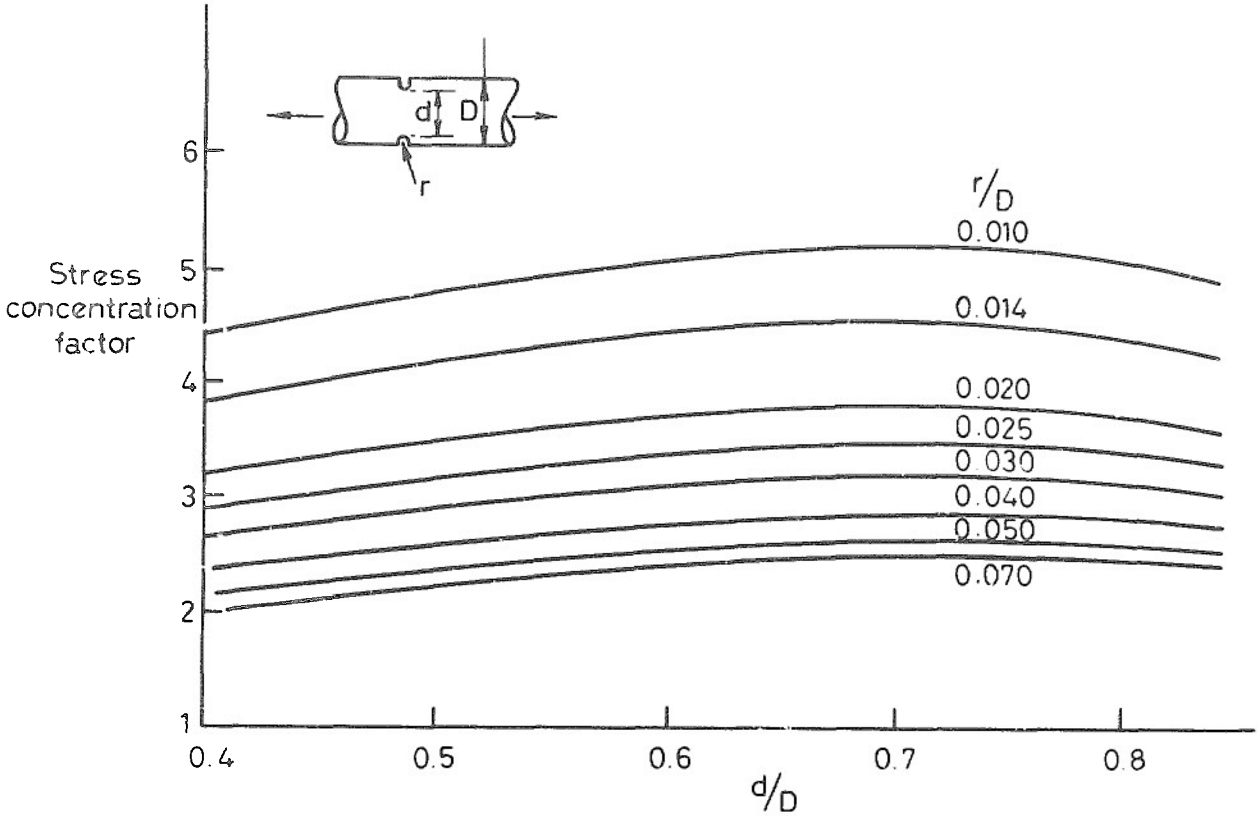


Figure 11. Calculated values of stress concentration factor (k_T) for a grooved shaft in tension (R.E. Petersen, Stress Concentration Factors, Figures 30 and 31).[27]

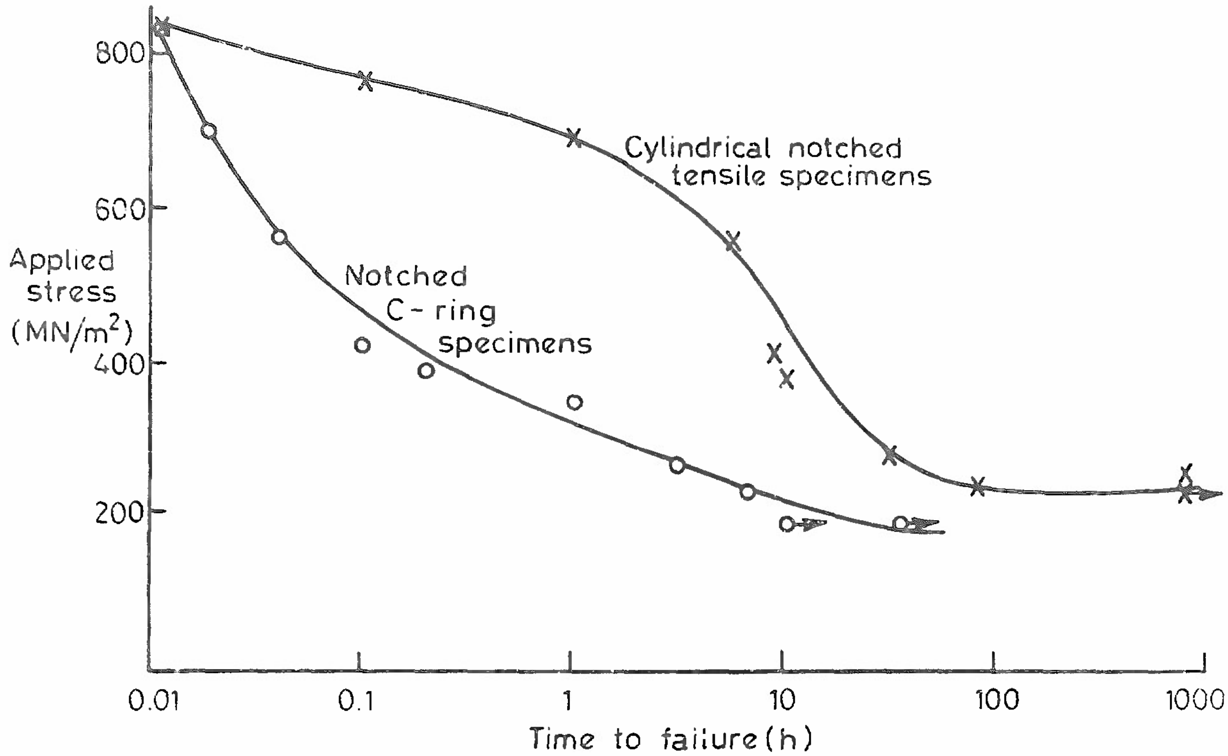


Figure 12. Comparison of time to failure of notched C-ring (k_T=4) and notched tensile specimens (k_T=6.3) chromium plated to a thickness of 0.05mm and loaded in air to various values of constant applied stress[28].

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16. Abstract

The present work demonstrates how a slow-strain rate tensile test can be used to quantify rapidly in a statistically rigorous manner the severity of hydrogen embrittlement produced in high strength 4340 steel by paint strippers and plating processes. The results of multiple slow strain rate tests conducted at a crosshead displacement rate of 2×10^{-4} mm/s using notched specimens in various paint strippers show that a minimum mean fracture stress and maximum standard deviation can be defined that correlates with the pass/fail criterion in standard notched C-ring tests. The advantages of using a slow strain rate test as a viable alternative to existing standard methods for hydrogen embrittlement testing are discussed.

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