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Hydrogen Embrittlement Susceptibility of Case Hardened Steel Fasteners

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Abstract

This work establishes the relationship between core hardness, case hardness, and case depth on susceptibility to hydrogen embrittlement of case hardened steel fasteners. Such fasteners have a high surface hardness in order to create their own threads in a mating hole, and are commonly used to attach bracketry and sheet metal in automotive applications. While case hardened fasteners have been studied previously, there are currently no processing guidelines supported by quantitative data for fastener standards. Through sustained load embrittlement testing techniques, the susceptibility of case hardened steel tapping screws to internal and environmental hydrogen embrittlement is examined. Further characterization of the fastener samples through microhardness testing, microstructure review, and fracture surface examination allows the investigation of susceptibility thresholds. It is shown that core hardness is the primary consideration for susceptibility. However, the fastener surface is prone to failure before the bulk section, up to the case depth, according to the case hardness. The zinc acid electroplating process used on the fasteners in this study appeared not to induce internal hydrogen embrittlement. However, baking durations commonly used for hydrogen embrittlement relief are shown to be ineffective and possibly detrimental.

Introduction

The susceptibility of case hardened fasteners to hydrogen embrittlement is not a new revelation. In 1996, Baggerly studied the failure of a heavy truck wheel bolt [1]. His failure and fracture mechanics analysis demonstrates that in such a high strength application, simply the act of installing a carburized fastener can create cracks in a case hardened surface. Additionally, once these cracks are formed, they create areas where localized corrosion can create an influx of hydrogen. In the bolts studied, this condition then led to fastener failure, with the failed bolts exhibiting a core hardness in the range of HRC 36 to 38 and a case hardness in the range of HRC 42 to 47. However, no conclusions are drawn as to the susceptibility of fasteners in less critical, high stress applications.

McCarthy, Wetzel, and Kloberdanz also studied the effects of hydrogen embrittlement in automotive fasteners with findings published in 1996 [2]. This study was quite comprehensive in scope, attempting to evaluate the effects of material, heat treatment, plating method, bake time, delay before baking, and others on the embrittlement of fasteners. The topic of interest to the current work was the study of case hardened 1022 steel. However, only one condition was studied, with a core hardness specified between HRC 28 and 36 and a case hardness specified as HRC 45 minimum. Three methods of testing were used to detect embrittlement, and to compare their effectiveness: a Chrysler plate test with sustained loading applied using a wedge under the fastener head specified as PS-9500, a General Motors bending test specified as GM-6661P, and a rising step load test. No failures were observed in the 1022 case hardened material, and therefore very few conclusions were drawn about its susceptibility.

As part of the study with McCarthy et al., Lukito and Szklarska-Smialowska evaluated the same materials for hydrogen trapping and permeability at Ohio State University [3]. Through their potentiostatic pulse experiments, they were able to evaluate a rate of hydrogen flux and trapping for the various materials studied. In comparing case hardened 1022 steel with 1022 steel that had been through hardened, it was shown that the case hardening reduced the hydrogen entry flux into the steel. It was therefore inferred that case hardening should reduce the susceptibility of 1022 steel compared to through hardening.

These studies demonstrate a lack of embrittlement susceptibility for the case-hardened condition evaluated and a possible positive effect of the case hardened layer. However, because only one condition was evaluated, minimal conclusions can be drawn as to the overall susceptibility levels of case hardened fasteners. Also, while the Ohio State study used hydrogen charging during slow strain rate embrittlement testing, the McCarthy et al. study did not consider the effects of environmentally induced hydrogen.

McCarthy and Shulke again addressed case hardened fasteners and hydrogen embrittlement in 2000, this time also considering the effects of environmental hydrogen sources [4]. In their study, they analyzed the performance of two common tapping screw materials: 1022 and 10B21 steel. In addition to using two materials, four heat treatment processes were compared: through hardening, neutral hardening, and the case hardening processes of carbonitriding and gas carburizing. Within each heat treatment group, the fasteners were subjected to a range of tempering temperatures, from as-quenched (no tempering) up to some groups tempered at 975°F. All groups were then embrittlement tested using the rising step load method with hydrogen charging. Based on the results of testing, several conclusions are drawn. The first is that the difference in composition from 1022 to 10B21 steel did not have an appreciable effect on susceptibility to embrittlement. Similarly, differences in performance between the two case-hardening methods were minimal and deemed insignificant. What was deemed significant was the effect of tempering temperature. A plot of embrittlement test results versus tempering temperature did indeed show a dramatic increase in performance as the tempering temperature increases up to 800°F. However, the study concludes that because the relationship is not linear, hardness is not a driving factor in susceptibility. The possibility that hardness could have a sigmoidal relationship with susceptibility, similar to the ductile to brittle fracture relationship with temperature is not addressed.

In 2002, the Industrial Fasteners Institute published a technical bulletin urging fastener manufacturers to limit the core hardness of many fasteners, including case hardened tapping screws, to HRC 36 or below [5]. The bulletin also states that the case hardness of those tapping screws did not appear to be nearly as influential as core hardness in embrittlement failures. However, the bulletin is based only on practical experience, and was not backed up with quantitative data from controlled experiments. A similar statement regarding core hardness can be found in the requirements listed for case hardened tapping screws in ASME B18.6.3. Section 4.8.1.1 on core hardness contains a statement reading "[core hardness] preferably should be no higher than Rockwell C36 to ensure against failure in assembly and service" [6]. However, this statement is again only a suggestion, and parts with a core hardness up to HRC 38 meet the standard.

Although the detrimental effects of case hardening on susceptibility to hydrogen embrittlement have long been recognized and specifically studied, little attention has been given to how processing may be improved or controlled to reduce the potential for failures. The ASTM F1941/F1941M-15 specification acknowledges a threshold for susceptibility of through hardened products, and prescribes baking treatments only for fasteners having hardness above HRC 39, validated by the results of research studies [7, 8, 9, 10]. However, it acknowledges no threshold for case hardened fasteners, and requires all case hardened fasteners to be baked and embrittlement tested regardless of hardness. If some of these fasteners are not susceptible, this is an inefficient and wasteful use of resources. If this work can show threshold levels below which hydrogen embrittlement failures should not occur, the industry may save time and resources, and end user risk may be reduced.

Methodology

Samples for Study

In order to establish material susceptibility thresholds, samples of fastener materials with different processing conditions, and consequently different microstructure and hardness parameters were required for testing. These were obtained by gathering multiple lots of tapping screws processed by a local heat treatment and plating facility. The fasteners used in this work were produced from either 1022 steel or 10B22 steel with boron and carburized to provide the required case hardness.

After heat treatment, the fasteners in this work all went through an electroplating process under similar conditions. The process used was a zinc-acid chloride barrel plating line; including an acid cleaning prior to plating. The standard practice for the facility is to bake the fasteners immediately after electroplating in an attempt to remove hydrogen absorbed during plating. For this work, samples were gathered after zinc electroplating, with one group of fasteners from each lot taken before the specified baking operation and another group taken after the baking operation. These groups are referred to in this work as batches. It is important to note that except for whether a batch was baked or not baked, the rest of the processing conditions within a lot are the same.

A summary of the fasteners provided for testing, along with the steel grade, the tempering temperature, and averages of the case hardness, case depth, and core hardness as measured by the processing facility, are shown in Table 1.

Test Methods

Fastener Sustained Load Embrittlement Testing in Air The first round of testing focused on using industry standard practices for detecting internal hydrogen embrittlement in tapping screws. All fasteners were tested in accordance with ASME B18.6.3, in a process that is known as a sustained load test. The intent of the test is to install the fasteners so as to apply a level of stress above the failure threshold, and then let the fastener remain in the stressed state for an extended period of time. If both the material microstructure condition (as indicated by the local hardness) and mobile hydrogen content thresholds are exceeded, failures should be observed. However, if no failures are observed, at least one of the thresholds must not have been exceeded. This test is meant to identify possible internal hydrogen embrittlement failures due to hydrogen present from any of the manufacturing processes. To perform the test, five screws are tightened to failure in a specified test plate. The fasteners are then installed to 80 percent of the average failure torque. Because only torque is being measured in this method, the actual tension, and therefore stress, on the fasteners is not known. However, based on tightening to 80 percent of the failure torque, it can be assumed that the fasteners are near their yield stress. After a period of time, the original tightening torque is reapplied. While ASME B18.6.3 prescribes that the original tightening torque be applied at 24 hours only, for this study a second application of torque was added at 48 hours.

In this round of testing, 19 batches were tested in the pre-baking condition, and 5 batches (from the same parent lots as 5 of the 19 batches) were tested in the post-baking condition. Each batch (pre or post) contained 15 fasteners.

Fastener Sustained Load Embrittlement Testing in Corrosive Environment For a given material microstructure state, the test variables that may be adjusted to evaluate susceptibility to hydrogen embrittlement in various possible service environments are: tensile stress and amount of mobile hydrogen. The second phase of testing focused on increasing the amount of mobile hydrogen available to initiate failures. To accomplish this, the ASME B18.6.3 testing was repeated, but with the addition of a corrosive environment during the two 24 hour periods between torque application.

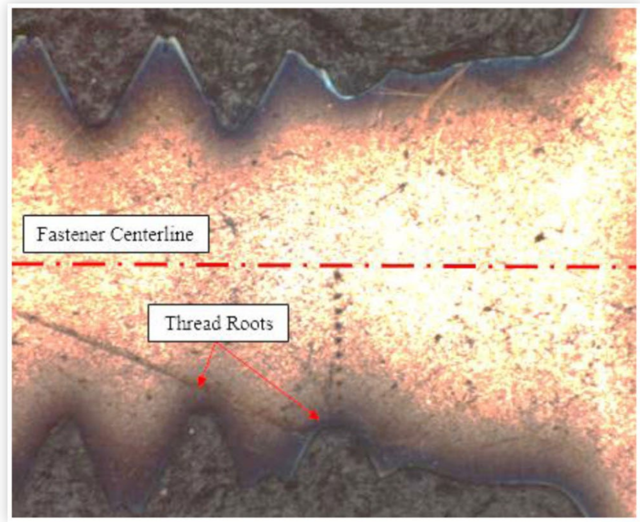
Fasteners were again installed into plates. The plates were then submerged in an approximately 1.75 wt% salt water solution and removed periodically and allowed to dry before being placed back into the solution. As before, the original assembly torque was reapplied at 24 and 48 hours. In this round of testing, 24 batches were tested, all post baking, again with 15 samples per batch.

Characterization of Hardness Profiles of Fasteners Tested Once fasteners were observed to either pass or fail embrittlement testing, the results were correlated with their internal microstructures. While many factors relate to the susceptibility of a material to hydrogen embrittlement, material strength is overwhelmingly pointed to as the most significant. In quench and tempered steel fasteners, hardness is used as a measure of strength.

The next phase of this study measured hardness profiles of the fasteners, in order to evaluate how far into the net section of the fastener the elevated surface hardness had some effect. Thus, several fasteners from each batch that underwent environmental embrittlement treatment and testing were measured. In lots that did not either pass or fail 100 percent, samples of both passing and failing fasteners were tested. The sample fasteners were sectioned longitudinally, mounted in phenolic resin, and polished for microhardness measurements. Attention was focused on the underhead fillets and thread roots of the fasteners where stress concentrations are highest. Starting at a thread root on each sample, Vickers hardness readings were taken at a distance of 0.002 inches from the surface, and every 0.003 inches travelling away from the surface towards the centerline, until the hardness gradient leveled off, with a final reading taken near the center of the fastener. An example of a typical hardness reading traverse is pictured in Figure 1.

Microscopic Examination of Fasteners Tested In addition to measuring hardness, the visual characteristics of the microstructures achieved during the case hardening process were also investigated. Beyond hardness, it was desired to examine the samples to evaluate if any significant differences in microstructure could be observed between fasteners that did or did not fail the environmental embrittlement testing. In order to capture a range of conditions, samples were chosen with high and low hardness, as well as samples that passed and failed the embrittlement testing. The fasteners

FIGURE 1 Typical microhardness reading locations (100X magnification)



and mounts used for microhardness testing were reused and etched to reveal microstructures. They were viewed at various magnifications, with attention paid to the surface state, and to how the microstructure changed as the material transitioned from the high hardness surface to the lower hardness core. The samples were also reviewed for signs of excessive inclusions or any other abnormalities that may affect susceptibility to hydrogen embrittlement failures.

To investigate the failure mode of the fasteners tested, failure surfaces were viewed both at low and high magnification via scanning electron microscopy (SEM). Fracture surfaces were analyzed in two samples: a fastener from batch 24 that failed in torsion while establishing assembly torques for embrittlement testing and a fastener from the same batch that failed during corrosive environment embrittlement testing.

In addition, several features observed during microstructure evaluation were observed in more detail via SEM. These were cracks observed both in samples that failed and samples that did not fail embrittlement testing.

Evaluation of the Zinc Electroplating Process The zinc electroplating process has many variables, such as the plating bath composition, applied current density, time spent in acid, and others. It has also been shown that many of these variables have at least some effect on the embrittling effects of the particular plating process. Further, it has been noted that passing the embrittlement tests in air could be due to any of the three threshold levels not being met. For this reason, it was desired to characterize the electroplating process used for producing the samples used in this study. ASTM F1940 prescribes a standardized method for quantifying the embrittling effects of an electroplating process utilizing the ASTM F1624 incremental step load (ISL) test procedure [11, 12]. For this testing, notched square bars made from alloy steel and hardened to HRC 52 are used as "worst case" witness samples processed along with actual parts through the full electroplating process. Each lot of bars is tested in the bare, preprocessed condition to establish a baseline strength. Bars are then ISL tested post-processing to determine the amount of embrittlement.

Of particular interest to this study was the effectiveness of the post plating baking operation. To evaluate this operation, notched square bars were processed with a lot of tapping screws on the same electroplating line used for the rest of the fasteners in this study. Four bar conditions were tested: one un-processed bar to establish a baseline, one bar that was removed after electroplating, but prior to baking, one bar that was electroplated and baked at the plating facility per their standard process for 11 hours at 200°C, and one bar that was removed after electroplating and baked in a lab furnace for 24 hours at 200°C.

Results

Fastener Sustained Load Embrittlement Testing in Air

In the first round of testing, 24 lots of 15 fasteners per lot were tested in air, after various processing treatments, according to industry standard practices. A total of 360 fasteners were evaluated, and no failures were observed. This lack of failures indicates that at least one of the hydrogen embrittlement variables (material microstructure, mobile hydrogen content, stress level, time), did not exceed the threshold to cause failures.

Fastener Sustained Load Embrittlement Testing in Corrosive Environment

The results of testing in a corrosive environment can be found in Table 2. Figure 2 shows the failures exhibited by Batch 19 tested in this fashion, which were typical of most failures observed. As shown in the Figure and results, 100 percent of the Batch 19 fasteners failed this testing in a corrosive environment, even though no failures were observed in the initial testing in air per ASME B18.6.3.

With failures observed, the results were plotted against case hardening variables in order to observe trends. Figure 3 shows the failure rate as a function of the average core hardness

FIGURE 2 Batch 19 after salt water embrittlement testing

FIGURE 3 Plot of failure rate versus nominal core hardness

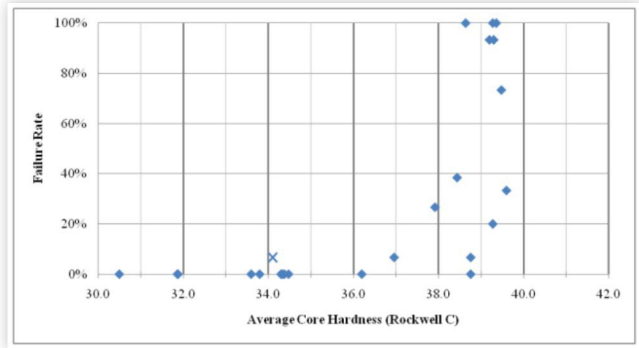
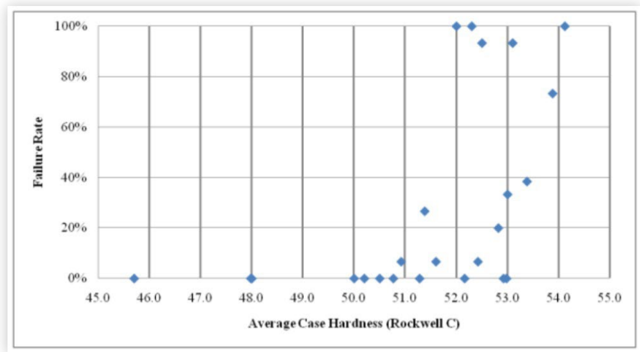


FIGURE 4 Plot of failure rate versus nominal case hardness



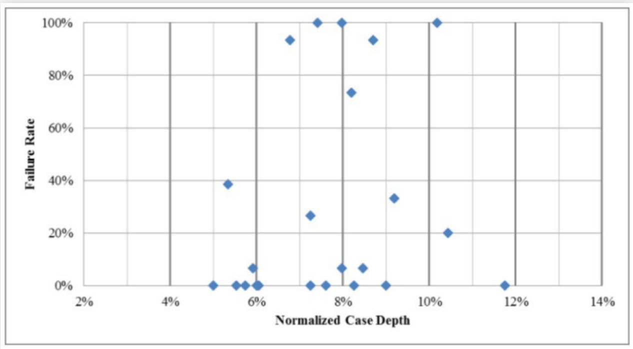
as reported by the heat treatment facility. It is important to note that the average core hardness represents a snapshot of the overall hardness of the lot, but not necessarily of the specific samples that failed. Due to the variation in hardness from sample to sample, the actual hardness varies by several (two to three) points on the Rockwell C scale. Figure 4 plots the failure rate versus the average case hardness as reported by the heat treatment facility, with the same considerations as mentioned for the core hardness.

Case depth is the first parameter that is affected by the geometry of the fastener. Clearly a case depth of 0.008 inches, for example, would have a much greater effect on a small diameter fastener than a large diameter fastener. For this reason, the average case depths measured by the heat treatment facility were normalized to create one relative scale. To normalize the values, the case depth was divided by the fastener radius at the thread roots. This value was then expressed as a percentage. Figure 5 plots the failure rate versus the normalized case depth.

Hardness Profiles of Fasteners Tested

The results of extensive microhardness testing of fasteners that both passed and failed the sustained load corrosive environment embrittlement testing can be found in Tables 3 and 4. Hardness readings of fasteners that passed embrittlement testing are shown in Table 3, while readings of fasteners that

FIGURE 5 Plot of failure rate versus normalized case depth



failed embrittlement testing are shown in Table 4. The "CORE" location indicates a reading taken near the center of the net fastener section. Figures 6 and 7 plot the hardness measurements of un-failed and failed samples respectively versus the distance from the surface. The "core" measurement was given a depth of 0.060 inches for the purposes of plotting.

Once again, because depth of hardness is expected to affect fasteners differently based on geometry, the data were normalized for further analysis. Figure 8 plots hardness versus a normalized depth for samples that failed embrittlement testing. The normalized depth is calculated by dividing the actual

FIGURE 6 Microhardness profiles of samples that passed embrittlement testing

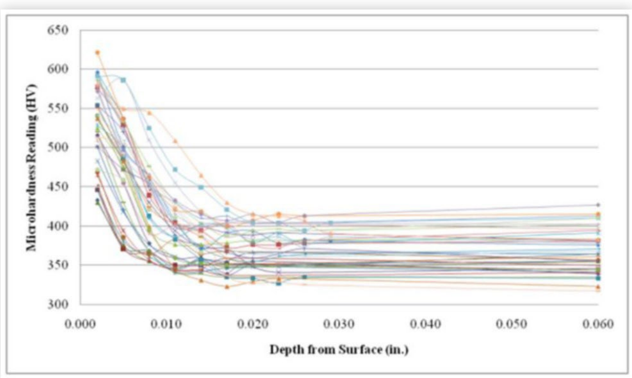


FIGURE 7 Microhardness profiles of samples that failed embrittlement testing

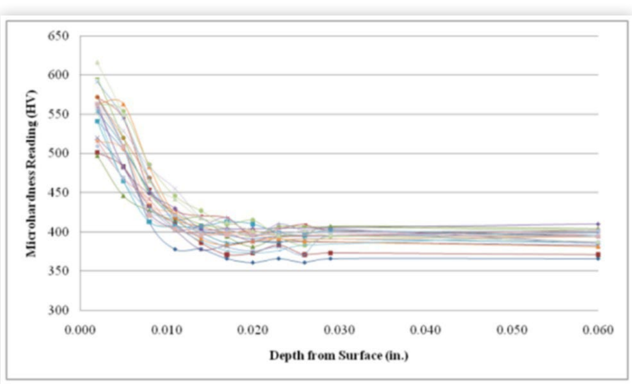
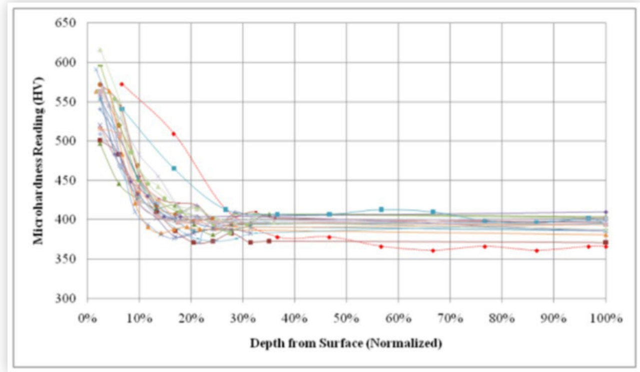


FIGURE 8 Normalized microhardness profiles of samples that failed embrittlement testing



distance from the surface by the fastener radius at the nominal minor diameter. In the case of the two samples measured across the washer feature, it is the actual distance divided by the net section width of the washer. These two samples stand out, as their thin section width causes a higher percentage of the cross-section area to be affected by the increased surface hardness.

Microscopic Examination of Fasteners Tested

An evaluation of the microstructure revealed predominantly tempered martensite in every fastener examined as expected. Sample images of both core and surface microstructures are seen in Figures 9 and 10. While some inclusions were found to be present, they were not extensive and were not evaluated further. The effect of the surface hardening process is evidenced in the change in appearance of the microstructure near the surface compared to the core. While still showing a structure of tempered martensite, the grain size appears smaller, and the additional carbides result in a much darker appearance from the increased carbon content during the carburization process.

The first fracture surface examined by SEM was that of a fastener from batch 24 that was failed in torsion, represented

FIGURE 9 Core microstructure showing predominantly tempered martensite at 1000X magnification

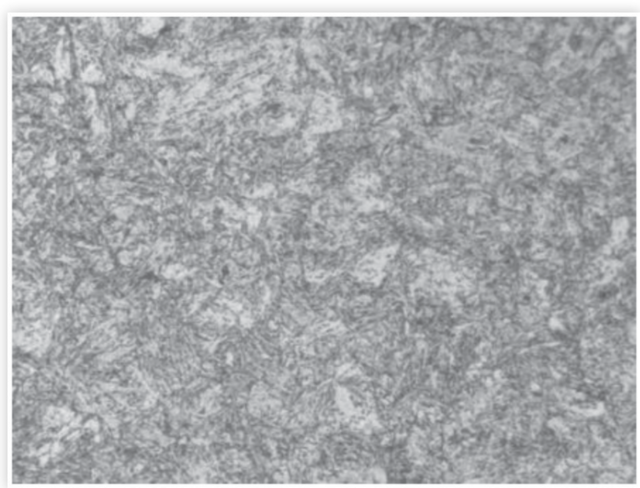
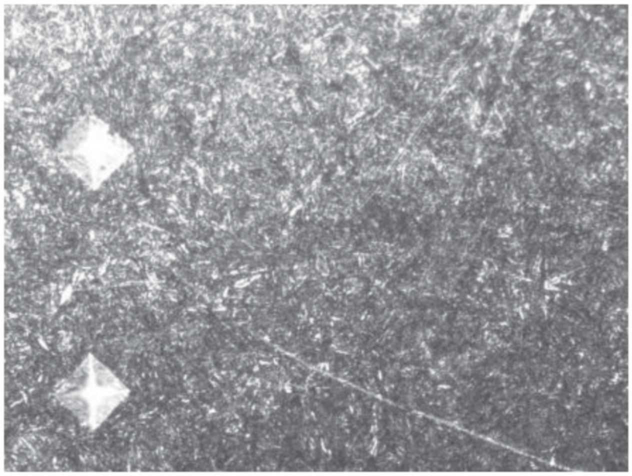


FIGURE 10 Near-surface microstructure showing predominantly tempered martensite at 500X magnification



in Figures 11 and 12. The fracture surface mainly showed evidence of ductile failure, except for the high hardness surface region, which showed evidence of brittle intergranular fracture. This evidence can be seen circled in Figure 12.

The next fastener examined was also from batch 24, but failed in sustained load embrittlement testing in a corrosive environment. Its fracture surface showed predominantly brittle intergranular fracture as seen in Figures 13 and 14. However, the lower hardness core also exhibited some ductile failure regions.

Next, a sample from Batch 30 that failed in corrosive environment embrittlement testing was studied further under SEM, because a crack of significant length was observed during microhardness testing. Figure 15 shows the location of typical failures as well as the additional crack observed. Although this was not the crack that caused the fastener to fail the embrittlement test, it may have contributed to significant loss of strength. As seen in Figure 16, it extends from what appears to be a "lap" in the material caused by the thread rolling process.

FIGURE 11 Torsion failure fracture surface - area of note magnified in Figure 12

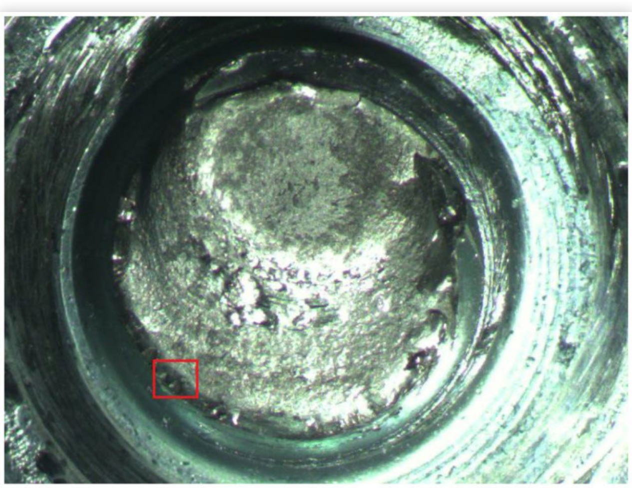


FIGURE 12 Torsion failure fracture surface at case hardened area 500X magnification

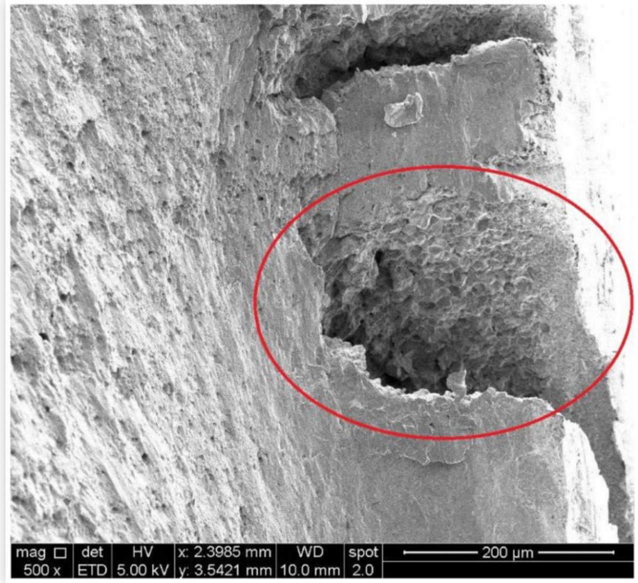
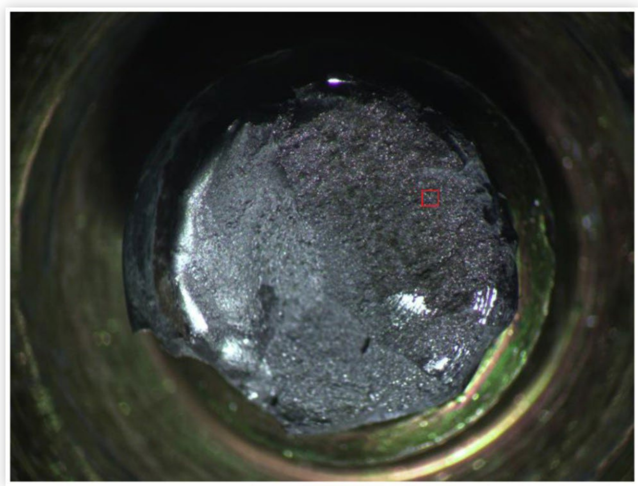


FIGURE 13 Corrosive environment embrittlement failure fracture surface - area of note magnified in Figure 14



More unexpected features were the cracks observed in a sample from Batch 17 that did not fail embrittlement testing. Figures 17 through 20 show multiple cracks extending a significant depth into the fastener core and a crack on the flank of a thread, which would not have been expected to be an area of highest stress concentration. In all cases, the cracks appear to be brittle intergranular in nature, progressing from the high hardness surface and ending in the lower hardness core.

Evaluation of the Zinc Electroplating Process

The zinc electroplating process has many variables, including: the number of baths, the composition of each bath, times

FIGURE 14 Batch 24 sample showing intergranular fracture 2000X magnification

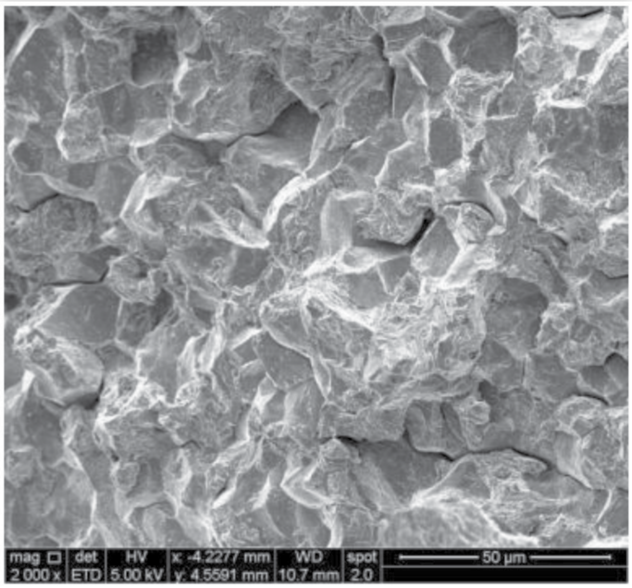
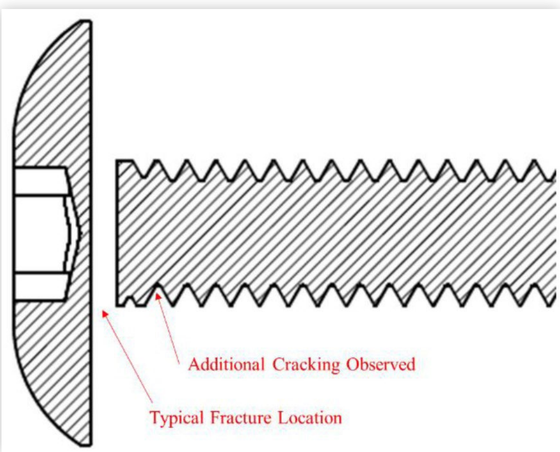


FIGURE 15 Schematic of batch 30 sample that failed embrittlement testing showing location of additional cracking



spent in each bath, and baking times and baking temperatures. The specific processing steps, times, temperatures and bath compositions were documented during the processing of ASTM F1940 notched square bars, and are provided in Table 5.

The average fracture loads measured during ISL testing of the bars are shown in Figure 21. Per the ASTM F1940 standard, if the fracture load of the plated bars exceeds 75 percent of the baseline value, the process is not considered embrittling. The sample not baked as part of the electroplating process fractured at a load that would not indicate it had been embrittled. However, both samples baked at 200°C fractured at values below 75 percent of the baseline value. This indicates that the inclusion of these baking processes actually contributed to embrittlement, rather than preventing it.

FIGURE 16 Batch 30 sample showing crack extending from thread lap 320X magnification

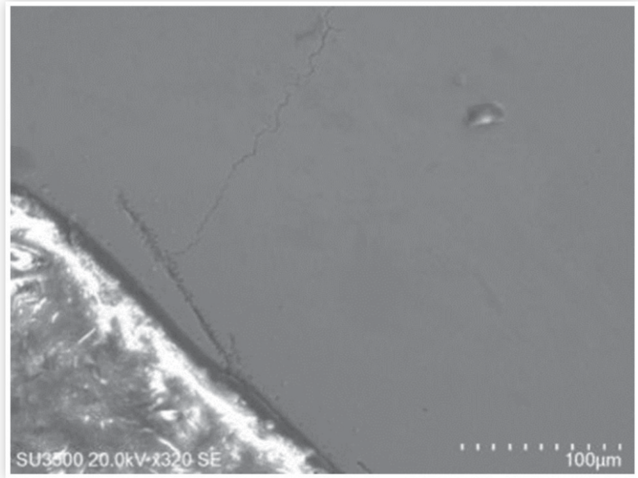
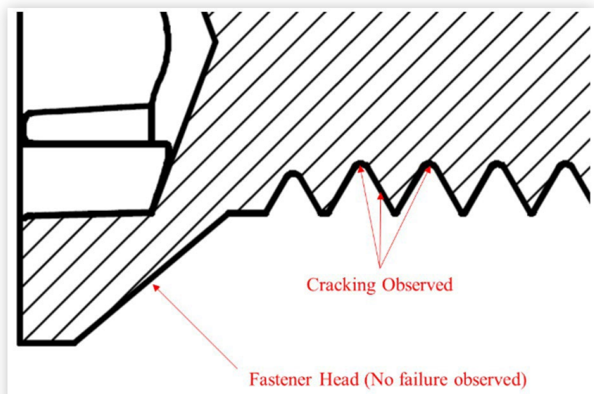


FIGURE 17 Schematic of batch 17 sample that passed embrittlement testing showing locations of cracks observed



Discussion

Fastener Sustained Load Embrittlement Testing in Air

According to ASME B18.6.3, tapping screws have a specified core hardness as high as HRC 38, and self drilling screws per SAE J78 have a specified core hardness as high as HRC 40 [13]. When combined with surface hardness of HRC 45 minimum and HRC 50 to 55, respectively, it was expected that a number of the samples collected would be susceptible to hydrogen embrittlement. In addition, the samples were all electroplated by a zinc acid process, which had been shown previously to be highly embrittling [10]. With these factors combined with samples that had not undergone any treatment for hydrogen embrittlement relief (baking), high rates of failures due to IHE from the process were expected. However, no failures were observed in any of the batches tested in air.

FIGURE 18 Crack in batch 17 sample that passed embrittlement testing 200X magnification

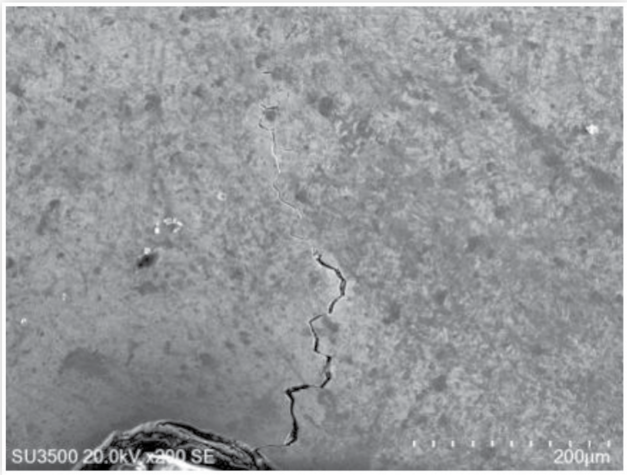


FIGURE 19 Crack in batch 17 sample that passed embrittlement testing 500X magnification

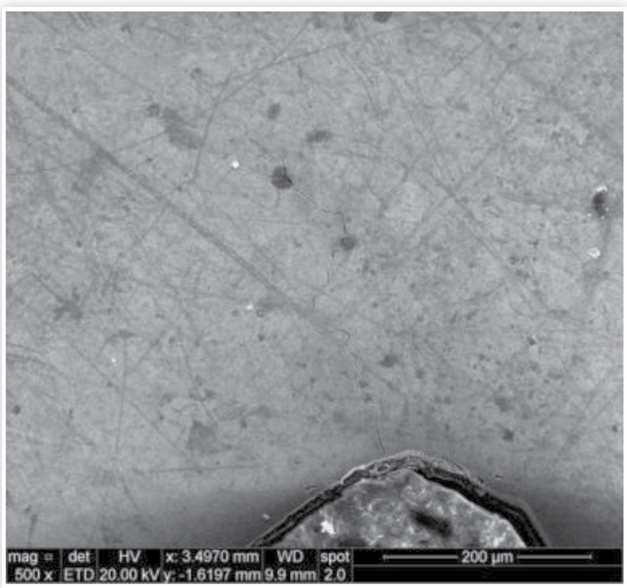


FIGURE 20 Crack in thread flank of batch 17 sample that passed embrittlement testing 500X magnification

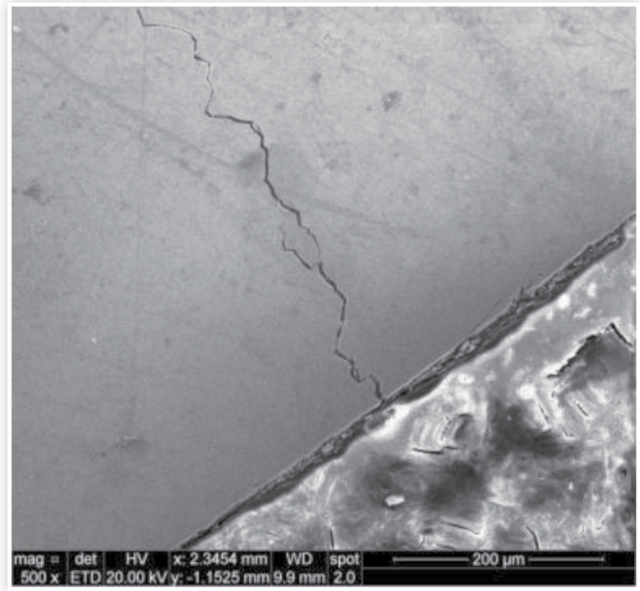
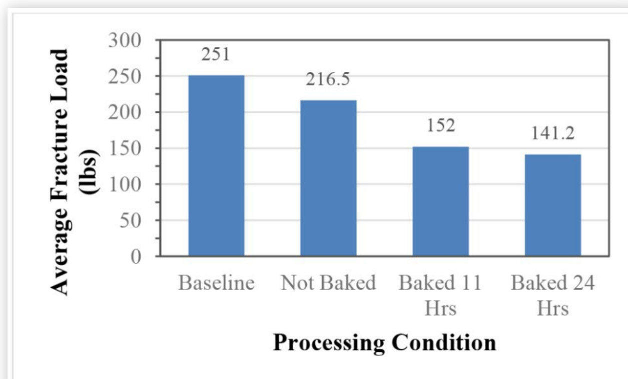


FIGURE 21 Results of ISL testing of notched bars



Rather than proving that the fasteners were not susceptible to hydrogen embrittlement, this result simply indicated that at least one, if not more, of the HE thresholds was not exceeded. Because the test was extended for 48 hours, the time to reach the failure threshold would have been expected to be exceeded. However, there are several possible answers as to why one of the other thresholds were not exceeded. These include: 1) not stressing the fasteners sufficiently, 2) insufficient hydrogen being present in the fasteners to cause failures, or 3) the fastener material conditions simply not being susceptible. While outside the scope of this paper, ASTM F1624 step load testing of actual fasteners was used to progressively increase the stress applied to the fasteners. Unlike the ASME B18.6.3 method, which is a pass/fail test, ASTM F1624 can quantify a stress at which embrittlement occurs by increasing stress slowly until failure. When tested in this manner the fasteners yielded rather than fracturing, indicating no signs of embrittlement. This showed that insufficient stress generated in the plate tests was not the reason for a lack of failures [14].

In general, the results of sustained load plate testing in air were deemed inconclusive. The lack of failures observed were likely due to a combination of several elements, and no conclusions on susceptibility thresholds or the effects of baking could be drawn from these results.

Fastener Sustained Load Embrittlement Testing in Corrosive Environment

The second phase of testing aimed to test samples after experiencing service conditions in which the threshold of mobile hydrogen content was exceeded. The idea was to provide sufficient hydrogen to ensure that lack of failure would indicate that either the material microstructure condition was safely below its critical threshold, or that the stress was below its critical threshold. By placing the plates of installed and stressed fasteners into a salt water solution, the corrosion process was the source of hydrogen. By definition, the addition of the corrosive environment made this a test mainly for environmental hydrogen embrittlement (EHE).

This test method produced failures in over half of the batches tested. Therefore the first conclusion drawn is that the sustained load plate test is capable of producing enough stress in the fasteners to induce failures when material susceptibility and hydrogen content thresholds are exceeded. However, it is still probable that the test would not be 100 percent effective in detecting embrittled fasteners, and more failures may have been observed if the stress was increased.

The more significant conclusion taken from this phase of testing is that fasteners meeting the hardness ranges specified in industry standards do exhibit material susceptibility. In through hardened fasteners, research has shown susceptibility to rise rapidly above a hardness of HRC 39 [9, 10]. If this threshold were to be applied to case hardened fasteners, either they would all be susceptible, as the surface hardness is well above HRC 39, or only a small portion would be susceptible as the core hardness is limited to HRC 38 or HRC 40 and below. The results of this round of testing indicate that the level of susceptibility lies somewhere in between. Failures in batches of fasteners with reported core hardness as low as HRC 37 indicate that the HRC 39 threshold does not apply when considering case hardened fasteners. However, the lack of any observed failures in fasteners with reported core hardness below HRC 37 would seem to indicate that the elevated surface hardness does not automatically put fasteners at risk. As with the testing in air, these results were again corroborated with ASTM F1624 step load testing of actual fasteners, this time with hydrogen generated through both a corrosive environment, and under an applied electric potential. With these additions of hydrogen charging, the time to failure and stress at failure were both quite low [14].

It should be noted that the corrosive environment the fasteners were exposed to in this test was quite aggressive. The distinction between internal and environmental hydrogen embrittlement must also be considered. Fasteners that fail this testing may still not be affected by IHE, or even EHE in a less corrosive environment. However, these failures were observed in a short timeframe, and an extended period in a less corrosive environment may yield the same results. In general, this study does not aim to comment on IHE or EHE in any certain real-world environments, but rather purely on material susceptibility.

Microscopic Examination of Fasteners Tested

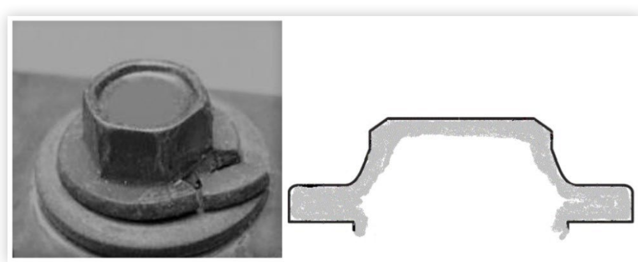
The embrittling nature of hydrogen was observed macroscopically when comparing samples failed dynamically in torsion and samples that failed the sustained load embrittlement testing. The torsional failures exhibited the expected ductile surfaces while the distinct transition to brittle intergranular fracture could be observed even without magnification when reviewing samples that failed embrittlement testing. This was further confirmed when viewing the fracture surface under high magnification via SEM. While some parts that failed embrittlement testing exhibited predominantly brittle intergranular fracture, some also showed areas of ductile failure. This is an indication that the entire material cross section would not have to be of a susceptible condition, but once the net section area is reduced by crack propagation, the fastener can fail due to excessive stress. This further emphasizes the importance of understanding the effects of case hardening and how much of the material may see an elevated hardness as a result.

Another feature observed during SEM analysis was the brittle intergranular fracture near the surface of a sample that was failed in torsion for establishing an assembly torque. Because it was not a delayed failure, the brittle fracture could not have been a result of hydrogen embrittlement. This feature is of interest, as during tightening the case hardened fasteners exhibited audible "popping" sounds as if they were beginning to crack well below the ultimate failure torque. If something in the nature of the high hardness outer case causes brittle cracks to form even during normal tightening, it may have the effect of increasing the susceptibility to EHE. This is another aspect that would be beneficial to evaluate further in future studies.

Much emphasis in this research was placed on determining the effects of case hardening on the cross sectional hardness profile of fasteners, and how the variables of case hardness, core hardness, and case depth interact. The first area to examine is the hardness readings of specific fasteners that failed or passed embrittlement testing. Microhardness testing corroborated the values reported by the heat treatment facility, with the lowest core hardness of any fastener that failed testing being read at 366 HV. This agrees well with the reported average core hardness of that lot of HRC 37. Thus, when considering the batches strictly by core hardness, the trend as plotted in Figure 3 indicates rapidly rising failure susceptibility as core hardness approaches and exceeds HRC 37. This is a significant finding in that it is two points lower on the Rockwell C scale than has been reported for through hardened fasteners [9, 10].

The outlying data point (plotted as an "X") in Figure 3 corresponds with the upper right-most data series plotted in Figure 8. This is a sample with a hex-washer head that failed through the thin washer section of the head, as seen in Figure 22, even though the fastener shaft did not fail or exhibit signs of cracking. This is another significant result in that it points to the geometry dependence of the susceptibility of case hardened fasteners to hydrogen embrittlement. If a case hardened fastener has locations of lower net section area, a larger percentage of the material is affected by the case hardening treatment. This

FIGURE 22 Failure in hex washer head of batch 14 sample (left) and representation of case hardened layer (grey areas, right)



is also depicted visually in Figure 22 and seen in Figure 8, where the hardness measured across the thin washer sections of two fasteners remained elevated nearly 30 percent of the distance into the section, as opposed to less than 20 percent in any of the threaded sections studied. In addition, if the fastener head is not parallel to the bearing surface, bending during installation can increase the applied stress. This would appear to be the primary influence of case depth on material susceptibility, as Figure 5 otherwise shows no correlation between failure rate and normalized case depth. Instead, geometry appears to be more influential than depth itself. However, it should also be noted that all samples tested had case depths conforming to specifications. Case depth exceeding specified values may greatly increase susceptibility to hydrogen embrittlement.

When considering the effects of case hardness, Figure 4 shows a similar correlation of increasing failure rate with increasing hardness as Figure 3. However, the relationship is less distinct than the trend with core hardness. Multiple batches with a case hardness near HRC 53 exhibited a zero percent failure rate, while multiple batches with a case hardness near HRC 52 exhibited a 100 percent failure rate. What does emerge though is a drastic reduction in failures as the case hardness drops below HRC 51. However, this is where the relationship between case hardness and core hardness should be examined. Figure 23 first plots both the nominal case and nominal core hardness for 24 lots in order of increasing core hardness. Figure 24 then plots the average case hardness as a function of the average core hardness, with a trend line plotted for reference. Both figures indicate that while it is not a direct correlation, in general when the core hardness increases, the case hardness will increase as

FIGURE 23 Case and core hardness of each lot tested

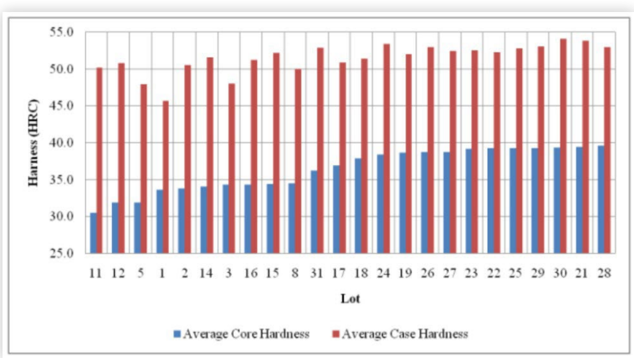
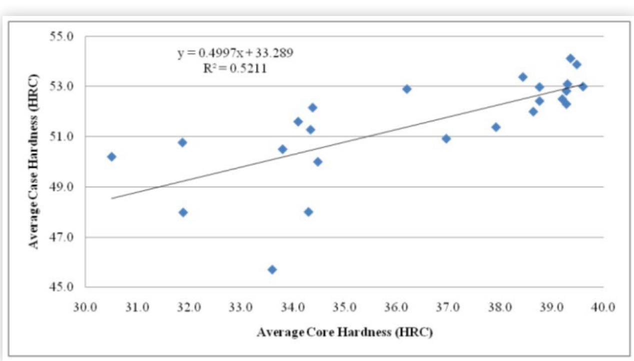


FIGURE 24 Relationship between case and core hardness

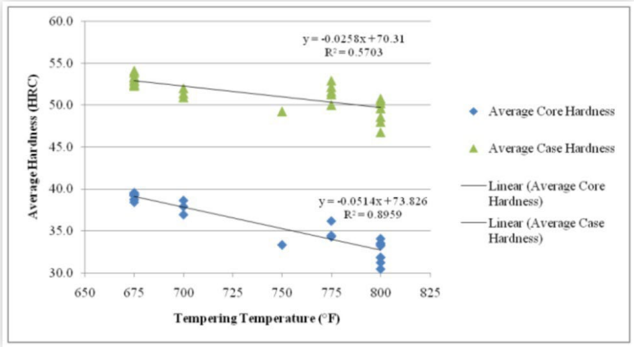


well. However, they also show that the case hardness increases at about half the rate. What this analysis indicates is that the two values cannot be considered independently. For example, the data would show that a core hardness of HRC 37 and a case hardness of HRC 45 would not be a realistic expectation to target during processing. Rather, to keep a high likelihood that the case hardness would be below the approximately HRC 51 threshold observed, a core hardness of approximately HRC 35 should be targeted. This is even more conservative than the HRC 37 threshold, but does appear to correlate well to the lack of failures observed in this range.

Because the two values are related, it is desirable to consider which value is more influential in the susceptibility of fasteners to hydrogen embrittlement failures. As noted, if the trend line plotted in Figure 24 is used to estimate core hardness, the case hardness threshold leads to a very conservative calculated core hardness threshold. However, when the observed core hardness threshold of approximately HRC 36.5 is used to calculate the case hardness, an accurate value of HRC 51.5 is obtained. This suggests that core hardness is the more significant indicator of susceptibility (and not case hardness). This finding would also appear to be supported by features observed during microscopic analysis. Figure 16 shows a brittle intergranular crack extending over 0.008 inches into the core of a failed sample, even though this crack did not cause the failure. As this fastener exhibited high hardness throughout, it is reasonable to believe that if the stress had been higher in the core region, the crack would have continued to extend through the entire cross section. However, Figures 18 through 20 show brittle intergranular cracks extending as far as 0.015 inches into the core of a sample from batch 17 that did not fail embrittlement testing. When compared with the microhardness readings, this is precisely the depth where hardness begins to drop to the HV 370 or HRC 37 range and below. These findings support the conclusion that the material condition at the surface of case hardened fasteners may always be susceptible to hydrogen assisted brittle intergranular cracking, but it is the condition of the core or bulk of the material that will ultimately determine if the fastener fails. Even if cracks begin in the high hardness surface, if the core is at a low enough hardness the crack will stop when it reaches the more ductile material. The fact that the threshold for core hardness is lower than that of through hardened fasteners exhibits the influence of case hardening in that it appears to create a condition much more likely to initiate cracks and allow them to grow a certain amount through the section. As the cracks grow, the net section area of the fastener is reduced, and the stress at the crack tip would be expected to rise. As stress increases, the material condition and hydrogen content thresholds required to propagate the crack would be reduced. Thus, case hardened fasteners create a much more susceptible condition than through hardened fasteners, with core hardness being the primary consideration on whether they are susceptible to failure.

If it is then determined that limiting core hardness below HRC 37 is the desirable end result of heat treatment, the method of ensuring this condition should be known. Fastener steel grade and property class standards such as SAE J429 and ISO 898-1 require a minimum tempering temperature to ensure proper material conditions [15, 16]. Figure 25 plots the

FIGURE 25 Effect of tempering temperature on hardness



reported average case and core hardness as a function of tempering temperature. SAE J78 on self drilling screws requires a minimum tempering temperature of 625°F, but from the fasteners in this study it would appear that a tempering temperature somewhere above 700°F would be required to ensure a core hardness below HRC 37. However, the effect of these temperatures on the ability to meet the minimum case hardness of HRC 50 for self drilling screws is unclear. As it was not the primary goal of this study, the effect of tempering temperature on material properties and hardness for various case hardening materials would need to be further investigated. However, the linear regressions plotted in Figure 25 reveal that core hardness is more affected by tempering temperature than is case hardness. From the viewpoint of processing to control hydrogen embrittlement susceptibility this is fortunate, as core hardness appears to be the primary variable to control. Additional investigations on the effect of tempering temperature on hydrogen embrittlement of fasteners should also consider and take care to avoid the possibility of the different but related problem of temper embrittlement.

Evaluation of the Zinc Electroplating Process

Because the zinc-acid electroplating process has been previously shown to be very embrittling, the results of sustained load testing in air showing no failures was unexpected. This made evaluation of the electroplating process used in this study a necessary step to gain more information on whether it was the process or the material making the fasteners resistant to IHE failures. If ASTM F1940 sampling showed the process to be embrittling to the standardized samples processed, it would show that the case hardened material may be more resistant. However, if the process was shown not to embrittle the samples processed, the effect of case hardened material would still not be evident in terms of IHE.

The results of ASTM F1940 sampling for this particular electroplating line were significant in two areas: the fracture strength of un-baked samples and the fracture strength of baked samples. As the process was expected to be embrittling, then notched bars that were processed with no baking step intended to remove hydrogen were expected to have low fracture strengths. The result of the sample processed in this way was the opposite, however, and would indicate that the process without baking did not introduce enough hydrogen to cause negative effects. Interestingly, the samples that were baked showed a reduction in fracture strength. This would indicate that in this case the baking process was actually detrimental to the performance of the parts. This is quite significant as much time and cost is spent on processing parts in this way under the assumption that it reduces the chances of embrittlement.

The question of baking effects is an extensive research project in itself, and is not further explored in this study. However, the findings in evaluating this process are another interesting data point. It should be noted that previous studies have shown that bake times of up to 24 hours or more may be required to restore the full strength of the notched bars used in this testing [17]. These findings seem to support that the 11 hour baking time used in this process is indeed insufficient, and in fact possibly detrimental. It also should be noted, however, that these results apply to the very high hardness notched bars tested, and the same results may not manifest in actual production parts.

Also, because the process without baking was not found to be embrittling even to worst case notch bars, the lack of failures in un-baked fastener samples is still inconclusive. It would appear that the process does not introduce sufficient amounts of hydrogen to cause failures, and it cannot be seen what the effects of the case hardened layer may be in this instance. When all of the tests are combined, no evidence of IHE caused by the process can be found, and this study can only comment on general material susceptibility of case hardened fasteners. It could be possible that with the same material conditions a different electroplating facility could introduce enough hydrogen to cause failures.

Summary/Conclusions

The primary goal of this study was to investigate the role of process parameters and surface hardness condition on the susceptibility of steel fasteners to hydrogen embrittlement. The material conditions investigated were core hardness, case hardness, and case depth. If threshold values for these conditions could be established, they could be used to inform and improve industry standards and practices in order to reduce or prevent hydrogen embrittlement failures in tapping screws. Multiple methods of hydrogen embrittlement testing were employed to investigate both IHE and EHE susceptibility. 24 lots of fasteners were processed under different conditions, tested, and extensively characterized leading to the following conclusions:

1. Case hardened material is susceptible to hydrogen embrittlement in the range of 37 to 40 HRC core hardness and 51 to 55 HRC case hardness, which falls within the hardness ranges currently specified in fastener standards for tapping screws such as ASME B18.6.3 and SAE J78.
2. Because of the hardness gradient from surface to core, failure susceptibility is geometry dependent (e.g. thin

sections such as found in washer head screws may fail even when the bulk section does not).

3. In the absence of thin sections, the data indicate a core hardness threshold of HRC 37, above which susceptibility to hydrogen embrittlement rises rapidly, as evidenced by a rapid increase in percentage of failures in sustained load testing in a corrosive environment.
4. Brittle cracking may occur in the higher hardness case of fasteners in this range, but it has been observed to stop once the lower hardness core is reached. Thus, it seems that the condition of the core is more important than the case in controlling failure.
5. Case depth within specified limits did not show any correlation with susceptibility to hydrogen embrittlement failures other than the effect on thin sections.
6. The combination of testing in air and in corrosive environments verified that fasteners that pass hydrogen embrittlement testing after processing (i.e. they do not suffer from IHE), may still be extremely susceptible to failure due to corrosion that generates significant amounts of hydrogen in use (EHE).
7. A minimum tempering temperature of 750°F may be effective in reducing hardness below threshold levels; however the effect of lowered hardness on tapping screw (namely SAE J78 self drilling screw) performance has not been verified.
8. Baking of fasteners for 8 to 12 hours shows no positive effects, and in fact shows a detrimental effect on worst case notched bars used to investigate the embrittling effects of the electroplating process per ASTM F1940.

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Appendix

TABLE 1 Fastener Samples Obtained for Testing

Lot	Fastener Description	Material	Tempering Temp. (°F)	Nominal Core (HRC)	Nominal Case (HRC)	Nominal Depth (in.)
1	6-32 x 3/8 Torx Pan TT Stl ZC	N/A	N/A	33.6	45.7	0.0025
2	6-32 x 3/8 Torx Pan TT Stl ZC	N/A	N/A	33.8	50.5	0.0030
3	6-32 x 3/8 Torx Pan TT Stl ZC	N/A	N/A	34.3	48.0	0.0040
5	8-32 x 3/8 Torx Pan TT Stl ZC	1022	800	31.9	48.0	0.0074
8	12-24 x .6 Ser HWH TT Dog Pt Stl ZC	1022	775	34.5	50.0	0.0060
11	10-24 x 1 HWH CA TT Stl ZC	1022	800	30.5	50.2	0.0040
12	M6 x 25 Torx Truss CA Tap-R Stl ZC	1022	800	31.9	50.8	0.0073
14	.370-12 x 1 HWH Type AB Stl ZC	10B22	775	34.1	51.6	0.0090
15	.370-12 x 1 HWH Type AB Stl ZC	10B22	775	34.4	52.2	0.0092
16	.370-12 x 1.25 HWH Type AB Stl ZC	10B22	775	34.3	51.3	0.0084
17	12-24 x 1.125 Torx Flat SDS Stl ZC	1022	700	37.0	50.9	0.0066
18	12-24 x 1.125 Torx Flat SDS Stl ZC	1022	700	37.9	51.4	0.0060
19	12-24 x 1.125 Torx Flat SDS Stl ZC	1022	700	38.6	52.0	0.0066
21	12-14 x 3/4 HWH SDS Stl ZC	1022	675	39.5	53.9	0.0066
22	1/4-14 x 3/4 HWH Crimptite SDS Stl ZC	1022	675	39.3	52.3	0.0070
23	1/4-14 x 3/4 HWH Crimptite SDS Stl ZC	1022	675	39.2	52.5	0.0064
24	5/16-12 x 1 HWH SDS Stl ZC	10B22	675	38.4	53.4	0.0064
25	12-14 x 1.25 HWH SDS Stl ZC	1022	675	39.3	52.8	0.0084
26	1/4-14 x 1.25 HWH SDS Stl ZC	1022	675	38.8	53.0	0.0078
27	1/4-14 x 1.5 HWH SDS Stl ZC	1022	675	38.8	52.4	0.0080
28	12-14 x 1.25 Torx Truss SDS Stl ZC	1022	N/A	39.6	53.0	0.0074
29	12-14 x 1.25 Torx Truss SDS Stl ZC	1022	N/A	39.3	53.1	0.0070
30	12-14 x 1.25 Torx Truss SDS Stl ZC	1022	675	39.4	54.1	0.0082
31	6-32 x .354 Torx Plus Pan TT Stl ZC	1022	775	36.2	52.9	0.0030

TABLE 2 Results of Sustained Load Embrittlement Testing in Corrosive Environment

Batch Number	24 Hour Failures	48 Hour Failures	Failure Rate (%)	Comments
1POST	0	0	0.00	
2POST	0	0	0.00	14 Tested
3POST	0	0	0.00	
5POST	0	0	0.00	
8POST	0	0	0.00	
11POST	0	0	0.00	
12POST	0	0	0.00	11 Tested
14POST	0	1*	6.67	Failure occurred in washer
15POST	0	0	0.00	
16POST	0	0	0.00	
17POST	0	1	6.67	
18POST	1	3	26.67	
19POST	15	N/A	100.00	
21POST	9	2	73.33	
22POST	15	N/A	100.00	
23POST	14	0	93.33	
24POST	4	1	38.46	13 Tested
25POST	3	0	20.00	
26POST	0	0	0.00	
27POST	1	0	6.67	
28POST	4	1	33.33	
29POST	10	4	93.33	
30POST	14	1	100.00	
31POST	0	0	0.00	

TABLE 3 - Microhardness profiles of Samples that Passed Embrittlement Testing

Batch - Sample No.	Hardness (HV) at Depth from Surface (in.)										
	0.002	0.005	0.008	0.011	0.014	0.017	0.020	0.023	0.026	0.029	CORE
1-1	433	373	368	346	359	352					355
1-2	446	371	366	350	350	352					344
2-1	430	376	355	346	357	348					355
2-2	537	483	430	394	373	371	348	341			346
3-1	446	381	361	341	341	346					364
3-2	469	386	364	383	364	368					357
5-1	501	421	368	344	357	355	366				364
5-2	452	373	355	344	344	359					350
8-1	520	430	368	341	339	335					335
8-2	516	455	378	359	359	339	355				339
11-1	554	486	413	383	357	335	333	327	335		333
11-2	537	483	399	346	331	323	329	333			323
12-1	483	418	376	361	348	337	352	348			341
12-2	465	394	357	344	344	337					341
14-1	541	472	394	359	352	350					344
15-1	554	520	452	388	376	366	359	364	371		355
15-2	596	537	459	404	373	359	355	361	364		371
16-1	550	490	424	402	386	359	352	352			361
17-1	596	501	462	407	371	391	394	378	383	381	376
17-2	577	529	439	404	394	373	376	376	378		381
17-3	525	476	396	376	376	378	381				383
18-1	501	472	452	399	399	391	388	391			381
18-2	529	497	410	386	402	399	378	391	378		391
21-1	621	537	462	421	418	399	407	415	413		415
23-1	586	525	455	430	410	413	404				413
24-1	586	537	446	402	394	402	376	371	381		394
24-2	586	533	476	430	404	386	394	396	394		402
25-1	572	497	465	433	415	407	415	404	413		427
26-1	591	586	525	472	449	421	404	404	394	404	410
26-2	572	596	537	497	459	418	394	399	391	381	394
27-1	581	550	545	509	465	430	415	413	407	391	383
27-2	563	586	509	455	418	396	383	386	373	386	378
28-1	509	455	391	402	399	404					396
28-2	472	459	421	410	366	388					410
29-1	577	490	455	415	399	404	407	404			402
31-1	541	509	452	421	388	371	364	364	361		368
31-2	513	505	465	424	391	337	327	329	325		317

TABLE 4 - Microhardness profiles of Fasteners that Failed Embrittlement Testing

	Hardness (HV) at Depth from Surface (in.)										
Batch - Sample No.	0.002	0.005	0.008	0.011	0.014	0.017	0.02	0.023	0.026	0.029	CORE
14-1 (Through Washer)	572	509	413	378	378	366	361	366	361	366	366
17-1	501	483	433	410	386	371	373	383	371	373	371
18-1	497	446	427	415	418	394	381	394	396	407	404
18-2	520	483	424	402	378	383	388	388			383
19-1	554	509	452	415	399	386	388	386	391		386
19-2	572	520	469	418	407	399	402	396			394
19-3	541	505	455	421	402	396	391	394			399
21-1	572	545	455	427	421	418	396	404	410	402	396
21-2	596	520	449	424	410	404	394	407	407		402
23-1	563	483	449	430	404	404	404				410
23-2 (Through Washer)	541	465	413	407	407	413	410	399	396	402	394
24-1	563	563	483	421	391	383	388	391	388		381
24-2	591	545	469	410	394	381	376	381	371	396	386
25-1	516	469	442	404	399	399	388	394			402
27-1	563	554	486	446	427	410	415	394	383	394	394
28-1	558	483	433	404	407	418	394	410	402	404	386
28-2	554	465	430	407	388	376	373	376	383		396
28-3	516	505	436	424	399	399	399	391			394
29-1	509	469	424	421	399	402	396	399			402
29-2	563	509	421	404	396	396	396				394
30-1	616	550	465	442	418	413	402	396	402	396	388
30-2	563	529	483	455	418	404	396	399	399	399	394

TABLE 5 - Electroplating Process Parameters

Step	Temp (°F)	Time	Comments
Soak Tank	148	15 min	3% sodium hydroxide solution with emulsifier package
Rinse 1	107	1 min	H2O
Rinse 2	85	30 sec	H2O
Acid Clean	96	15 min	20 degree Baume Muriatic acid (HCL) mixed with water to make the tank a 32.5% acid concentration mixed with 2% inhibitor.
Rinse 3	87	1 min	H2O
Rinse 4	83	30 sec	H2O
Rinse 5	74	2 min	H2O
Electro Clean	151		10% sodium hydroxide solution
Rinse 6	112	15 sec	H2O
Rinse 7	89	2 min	H2O
Rinse 8	83	30 sec	H2O
Electroplate	92	7652 sec (127.5 min)	800 Amps, wetter, brightener, boric acid, potassium chloride and Muriatic Acid (HCL) plating efficiency of the bath is 98%
Rinse 9		Dip	H2O
Rinse 10	75	2 min	H2O
Rinse 11	83	15 sec	H2O
Rinse 12	66	3 min	H2O
Acid Etch	72	10 sec	
Chromate	78	15 sec	Clepo PK
Hot Rinse	117	30 sec	H2O
Bake	403	668 min (11.1 hrs)	

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