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Influence of De-icers on the Corrosion and Fatigue Behavior of 4140 Steel

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The purpose of this test was to evaluate the effects of calcium magnesium acetate (CMA) and sodium chloride (NaCl)—two common substances used to de-ice roadways—on the corrosion and fatigue behavior of annealed AISI 4140 steel. When CMA-corroded, NaCl-corroded, and as-machined samples were tested using $R = 0.1$, and $f = 20$ Hz, it was found that, within the scope of this study, samples corroded in both 3.5% CMA solution and 3.5% NaCl solution exhibited a lower fatigue strength than samples tested in the as-machined, uncorroded condition. For the short lives tested in this study, the difference in the effects of CMA and NaCl is minimal. However, at longer lives it is suspected, based on the trends, that the CMA solution would be less detrimental to the fatigue life.

Keywords automotive, carbon/alloy steels, corrosion testing, mechanical testing

1. Introduction

Calcium magnesium acetate (CMA) and sodium chloride (NaCl) are two common substances used in de-icing roadways. As seen in cold climates, road salt can be very corrosive and destructive to automotive components. While it has been cited in some sources that CMA is less harmful to the environment than NaCl (Ref 1), the effects of CMA and NaCl corrosion on fatigue strength have not been compared or quantified. Furthermore, automotive parts are subjected to many types of cyclic loading, so the effect of corrosion on fatigue strength of these components is vital information in part design. Annealed AISI 4140 steel is a chromium steel alloy commonly used in automotive parts, such as spindles, axles, and clutch components (Ref 2). These components are subjected to many repeated loads; so a spindle, axle, or clutch that is exposed to a corrosive environment must be able to withstand corrosion, and the fatigue strength of the steel must not be compromised.

In order to test the effects of both CMA and NaCl on the fatigue strength of annealed AISI 4140, two corrosion baths were prepared to simulate an environment similar to one found on an automobile exposed to highly corrosive environments—one bath simulating an environment in which NaCl was used as a de-icer, and the other bath mimicking a similar CMA-based environment. The samples were corroded for the same length of time and in the same manner. Upon completion of the corrosion process, tensile fatigue tests were performed. Several "as-machined" samples were also tested as a control. The details of the experiment are in the following section.

2. Experimental Procedures

The chemical composition of AISI 4140 is detailed in Table 1. The chromium present in AISI 4140 is vital in resisting corrosion. Sixteen tensile samples were machined from 12.7 mm (0.5 in.) diameter rod stock of annealed AISI 4140. No surface roughness measurements were made of the as-machined samples. Therefore, the detailed parameters used to machine the samples are provided. The specimens were machined using a computer numerical controlled (CNC) lathe. The cut speed was 75 m/min (246 surface feet per minute). A feed rate of 0.1524 mm per revolution (0.006 in. per revolution) was used, as well as a depth of cut (DOC) of 0.0762 mm (0.003 in.). Each specimen received a 0.254 mm (0.001 in.) finish pass, and one spring pass. A Kennametal Top Notch tool holder was used with NR3047R (1.1913 mm full radius) grooving/turning inserts, made of an uncoated tungsten-carbide (grade SC23).

The fatigue test samples had a test section with a length of 31.75 mm, which had constant a circular cross section with a diameter of 6.35 mm. Sixteen samples were machined, and the following test plan was followed:

- Six as-machined samples
- Five CMA-corroded samples
- Five NaCl-corroded samples.

For the corrosion process the machined portion of the samples were cleaned with alcohol to remove any oils or debris. Next they were divided into two groups of five and placed into two separate racks. Two polymer containment vessels were filled with 5 L of water, each. A 3.5% concentration was achieved by mixing 175 g of NaCl and CMA in to separate baths. The samples were placed horizontally in a holding rack (shown in Fig. 1). Once the NaCl and CMA had fully dissolved, the

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Table 1 Chemical composition of annealed AISI 4140 steel

Carbon, C	0.380-0.430%
Chromium, Cr	0.80-1.10%
Iron, Fe	96.785-97.77%
Manganese, Mn	0.75-1.0%
Molybdenum, Mo	0.15-0.25%
Phosphorous, P	≤0.035%
Silicon, Si	0.15-0.30%
Sulfur, S	≤0.040%
Yield strength, MPa	415

Source Song and Shieh (Ref 4)

Fig. 1 Samples in holding rack prepared for immersion in the corrosion bath

samples were lowered into the corrosive solution and the top of the vessels was covered. Aerators were used to keep the solutions circulating and to keep NaCl and CMA solute from settling out of the solution. The corrosion tanks were kept at room temperature and out of direct sun light.

Five samples were submerged in the NaCl solution and five in the CMA solution. The samples were soaked in the corrosive solutions, and removed after an average of 13 h. The samples were then dried in air for 15 min, to provide a moist oxygen-rich environment, as would be expected on automobile components, before being placed back in to the solution for another 13 h. This was repeated for 18 cycles. The total time the samples soaked in the baths was 232 h and 55 min, and the total time the samples were out of the baths was 4 h.

Fatigue testing was performed on a 100 kN MTS servohydraulic test machine. A stress ratio, R, of 0.1 was used, per Koshnaw et al. (Ref 3), and Song and Shieh (Ref 4). A frequency of 20 Hz was used. Before beginning tests, the grip sections of the samples were cleaned with steel wool to prevent both slipping of the samples during testing and damage to the machine.

3. Results

As shown in Fig. 2, the corrosion progressed rapidly over the course of 9 days. The corrosion caused by the NaCl penetrated deeply into the surface of the samples causing pitting. The corrosion caused by the CMA appeared to be mostly confined to the surface, and there were also no signs of pitting or loss of area due to this oxidation. The CMA reacted with the steel in such a way that the oxide layer appeared to protect the steel from further oxidation, but more analysis needs to be performed to validate this claim.

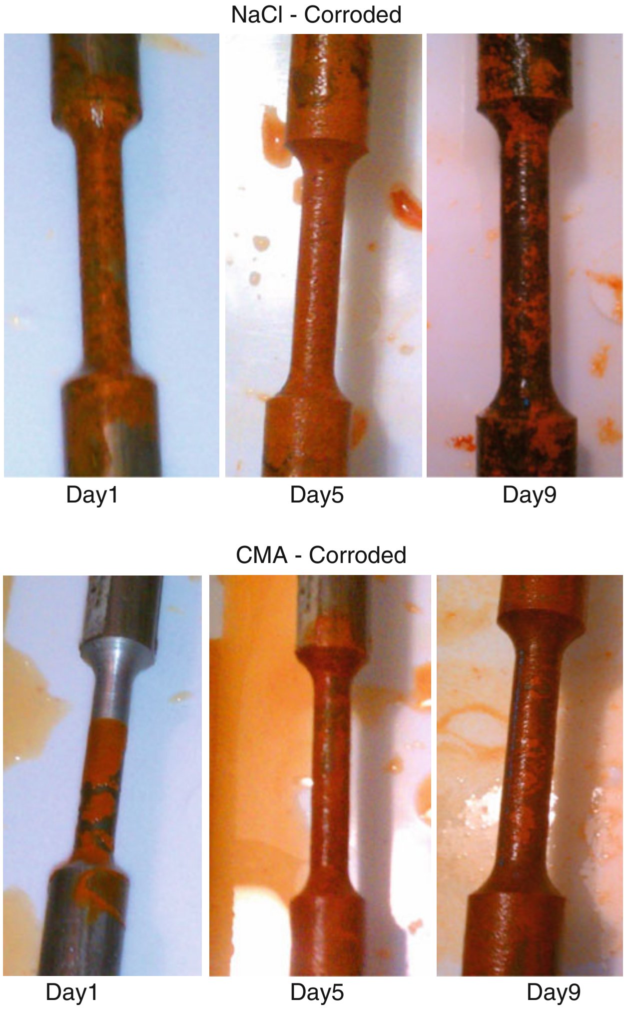

Fig. 2 NaCl and CMA samples throughout the corrosion process

As previously stated six samples were left as-machined for fatigue testing. The ten remaining samples were corroded in the baths using the methods described above. The as-machined samples, as well as the results of the corrosion can be seen in the images shown in Fig. 3.

The test parameters and numbers of cycles until failure for each sample are shown in Table 2. This S-N curve is presented in Fig. 4. The as-machined samples had longer lives at all stress levels except at the highest stress. At the highest stress it is not expected that superficial surface damage would control life. The highest stress level (~75% of reported yield strength) was high enough to actually provide some localized yielding, as was seen by a reduction in cross-sectional area. The variation in fatigue life for the three test conditions at the highest stress is more than likely a statistical phenomenon and not a direct function of the surface condition. The two corrosion baths had very little difference between the two for the stresses tested. The trend however, based on these results, is for NaCl to be more detrimental to the fatigue life. The lowest stress resulted

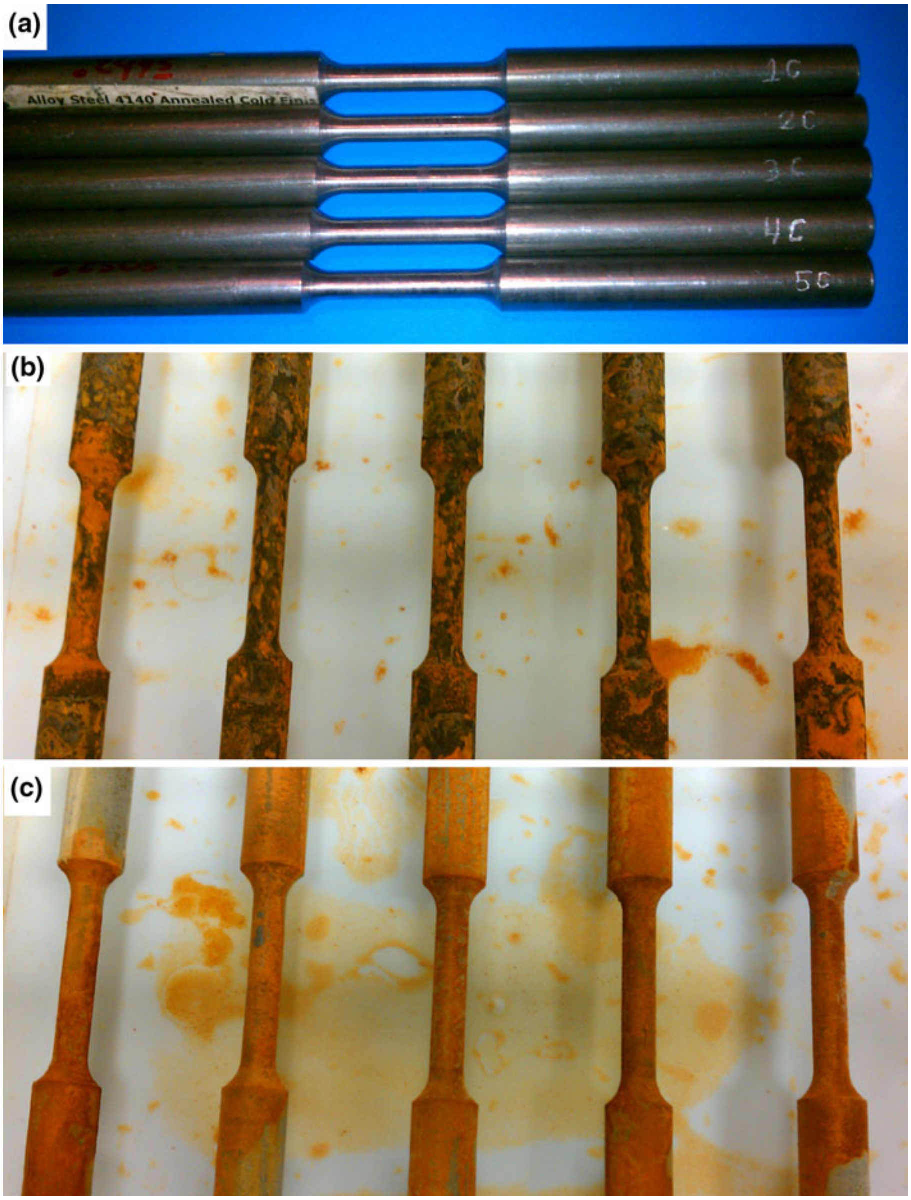

Fig. 3 Fatigue test samples are shown in the (a) as-machined condition, (b) NaCl-corroded condition, and (c) CMA-corroded condition

in runout tests of 10^6 cycles for all conditions (those data points are not shown in Fig. 4 for simplicity).

Figure 5 shows the fracture surfaces of each sample that ran until failure, as well as the maximum stress that was applied to the sample. These images were taken a few days after completing all the tests. The fracture surfaces of the as-machined samples and the samples that were exposed to CMA appear the same as they did immediately after testing. The samples subjected to the NaCl bath have fracture surfaces that oxidized after testing, and possibly somewhat during testing as well.

4. Discussion

The CMA solution has been previously shown to have a lower corrosion rate than NaCl (Ref 1). This was expected in this study as well. As previously mentioned the samples exposed to CMA were not pitted or corroded as dramatically as were the samples immersed in the NaCl solution. After fatigue testing the fracture surfaces were visually inspected. After a few days the fracture surfaces of the samples exposed to NaCl had oxidized, but those subjected to CMA had not. The indication is that moisture containing NaCl was still possibly present and trapped in the irregularly shaped oxide surface that formed as a result of the NaCl exposure. The oxide was fractured during the fatigue test allowing the fracture surfaces to be exposed to the once trapped moisture. At longer lives this could even influence fatigue crack growth. If some sort of moisture is present in the oxide, then it could also penetrate to the crack tip accelerating the crack growth rate. This effect was not directly evaluated in this study, and is being proposed, at this stage, only as a possible contributing mechanism that could influence the fatigue life.

As expected, the fatigue strengths of both corroded samples were significantly lower than the as-machined samples. At

Table 2 Number of cycles for each stress state for the as-machined, NaCl corroded, and CMA-corroded fatigue test samples

Sample #	Sample_0	Sample_1	Sample_2	Sample_3	Sample_4	Sample_5
As-machined						
σ_{Max}, MPa	400	550	615	700	650	510
σ_{Min}, MPa	40	55	61.5	70	65	51
σ_{Mean}, MPa	220	302.5	338.25	385	357.5	280.5
$\sigma_{amplitude}$, MPa	180	247.5	276.75	315	292.5	229.5
P_{mean}, kN	6.97	9.58	10.71	12.19	11.32	8.88
$P_{amplitude}$, kN	5.70	7.84	8.76	9.98	9.26	7.27
N-cycles	1,000,000	198,087	83,839	1889	45,908	325,411

Sample #	Sample_1	Sample_2	Sample_3	Sample_4	Sample_5
NaCl corroded					
σ_{Max}, MPa	615	700	510	550	400
σ_{Min}, MPa	61.5	70	51	55	40
σ_{Mean}, MPa	338.25	385	280.5	302.5	220
$\sigma_{amplitude}$, MPa	276.75	315	229.5	247.5	180
P_{mean}, kN	10.71	12.19	8.88	9.58	6.97
$P_{amplitude}$, kN	8.76	9.98	7.27	7.84	5.70
N-cycles	76,007	17,882	162,625	95,053	1,000,000

Sample #	Sample_1	Sample_2	Sample_3	Sample_4	Sample_5
CMA corroded					
σ_{Max}, MPa	700	615	550	510	400
σ_{Min}, MPa	70	61.5	55	51	40
σ_{Mean}, MPa	385	338.25	302.5	280.5	220
$\sigma_{amplitude}$, MPa	315	276.75	247.5	229.5	180
P_{mean}, kN	12.19	10.71	9.58	8.88	6.97
$P_{amplitude}$, kN	9.98	8.76	7.84	7.27	5.70
N-cycles	4288	48,191	73,235	197,118	1,000,000

N-cycles that are equal to 1 million were runout samples

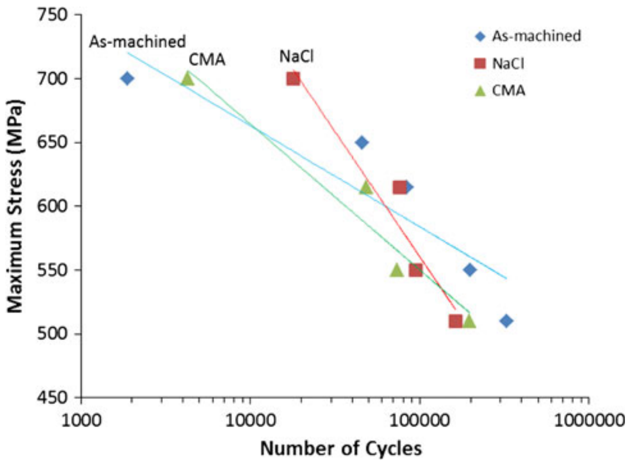

Fig. 4 Maximum stress versus number of cycles until failure

medium stress ranges (550 and 615 MPa) CMA exhibited the lowest fatigue strength. At lower stress (510 MPa) the NaCl sample exhibited the lowest strength; at a point below the yield strength (400 MPa) all three samples were runouts and were stopped after 10^6 cycles. This is based on the endurance limit defined in *Machine Component Design* (Ref 5). At 700 MPa the as-machined samples exhibited the lowest fatigue strength, although the differences between the three conditions, as was explained previously, were probably a result of statistical variations (although the scope of this study did not allow for evaluating the statistical variations in the mechanical behavior, nevertheless it is being proposed as a possible cause for the results at the highest stress). At the higher loads the surface conditions have a smaller influence on the fatigue life. As seen by the plot in Fig. 4, the *S-N* curves for CMA and NaCl trend below that of the as-machined samples at the longer lives, as would be expected since, in the longer life regime, the life is dominated by nucleation, which is typically controlled by the surface conditions. The *S-N* curves were developed using a logarithmic curve fit. Based on the plot, CMA-corroded samples exhibit a lower fatigue strength than both those corroded in NaCl and the as-machined samples, specifically at higher stress levels. However, the trend of the data is that the samples subjected to the NaCl bath would have shorter fatigue lives than the other two conditions at the longer life region of the curve; however that was outside the scope of this study. This would be expected based on the amount of corrosion seen on the samples exposed to NaCl versus that present on the samples submerged in the CMA solution.

It was observed from this study that the NaCl-corroded samples showed much more visible corrosion over time. After the testing was complete, the samples were stored in the testing lab. Two weeks after the testing was complete, it was visually apparent that the NaCl-corroded samples continued to corrode. The CMA-corroded samples did not seem to continue to

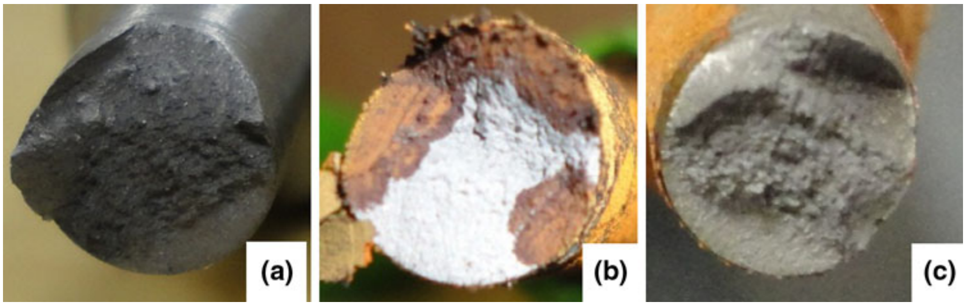

Fig. 5 Fatigue fracture surfaces of (a) as-machined (max stress = 650 MPa), (b) NaCl-exposed (max stress = 550 MPa), and (c) CMA-exposed (max stress = 615 MPa) samples. The NaCl fracture surface exhibited corrosion after the fatigue test was completed indicating moisture and NaCl were trapped in the oxide surface layer

corrode, upon visual inspection. Conversely, the NaCl-corroded samples seemed to corrode further; the corrosion even seemed to spread to the clamp sections, whereas the CMA-corroded samples had corrosion that was mostly contained within the machined test section. Furthermore, when corrosion flakes came off of the samples, the CMA-corroded samples showed bare metal; the NaCl-corroded samples showed that the corrosion had further penetrated the samples, turning them a blackish color. This excessive post-bath corrosion shows that short-term and long-term corrosions may very well produce different results.

As such, further testing should be done to better quantify the results. The specimens were subjected to a short-term corrosion bath, but a longer corrosion period would be more ideal for the study of the effect of corrosion on fatigue strength. Vehicle components are not only subjected to corrosion while in direct contact with the corrosive substance but also, as time passes, the corrosion spreads and worsens as the material is exposed to oxygen in the air. It is possible that one substance will have more of a substantial long-term effect on the fatigue strength of the vehicles' components than another. In the pictures it is noticeable that the samples that were corroded in NaCl solution continued to corrode much more intensely than that of the CMA solution. For a more thorough study, it is recommended that a long-term approach be followed.

5. Conclusions

The following conclusions were made from this study.

1. The NaCl solution was a more corrosive medium than the CMA solution based on a visual inspection.
2. Corrosion from the NaCl solution continued even after the samples were removed from the bath and fatigue tested, with the fracture surface exhibiting evidence of corrosion.
3. The as-machined, uncorroded samples had the longest fatigue lives at the stress levels tested. At some loads the samples exposed to CMA had the shortest life, but the trend was for the fatigue lives to be shortest for the samples immersed in NaCl at the higher cycle test region.

Acknowledgments

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