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**Environmentally Assisted Cracking Concerns
for Cadmium Replacement**

by Scott M. Grendahl

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Environmentally Assisted Cracking: Concerns for Cadmium Replacement

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14. ABSTRACT Recently, cadmium replacement has become the topic of an Environmental Security Technology Certification Program. As a direct result of this program, replacement coating systems will be developed and scrutinized. A fundamental starting point for corrosion-resistant coatings development work consists of a target for the overall chemical potential (galvanic series) and pH of the coating system and the substrate. This work developed the data necessary to determine the target for several high strength steels. Hydrogen is widely recognized as having a detrimental embrittling effect on high strength materials, especially high strength steel. Many of these materials show a strong dependence of hydrogen on chemical potential and pH. This work focused on determining the effect of hydrogen on the environmentally assisted cracking critical stress intensity values (K_{IEAC}) for several common high strength structural steels. Evaluating the effects of hydrogen on K_{IEAC} over a range of chemical potential and pH will make the optimum chemical potential and pH evident for each material. It then becomes possible for the coating system designers to tailor a prospective coating system around those optimum values with the various substrates. If a system were designed at the optimum values for chemical potential and pH, the substrate material would be at the most tolerant conditions for resisting detrimental hydrogen effects, regardless of the source of the hydrogen.				
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1. Introduction

Cadmium coatings are currently used in every major land, sea, and air defense system in a wide variety of system-critical applications. Cadmium plays an important role in the substrate-coating relationship. The functional performance of the coating system contributes to the longevity and mechanical performance of any coated component. In the case of high strength steels, the functional performance of cadmium coatings as part of the substrate-coating system evolved over several decades of in-service use. Because of the wide range of environmental regulation and costs associated with the use, handling, and disposal of cadmium-containing components and processes, the Government has determined that a replacement must be found. A fundamental starting point for the research required to accomplish this task begins with the determination of the materials that are common substrates for the cadmium-containing coating systems.

These materials, most commonly high strength steels, are deleteriously affected by hydrogen generated at or near the surface from the natural corrosion process of the environment, particularly when they possess yield strengths above 1400 MPa (1). This deleterious effect of hydrogen on high strength material by the environment is typically referred to as environmentally assisted cracking (EAC). Failures of military grade steels with cadmium-containing coating systems have been reported extensively in the literature (2–7). Since all substrates reach equilibrium in the environment (in terms of pH and electrochemical potential) with their respective coating systems, it would be extremely beneficial to know the optimum pH and electrochemical potential where the tolerance of the high strength material to EAC is at the maximum level. A coating system could then be designed around those parameters for each individual material. If a coating system were designed with no regard for the equilibrium pH and electrochemical potential, it is quite possible that the system could fail prematurely, since high strength materials demonstrate a strong dependence on these parameters. Although there are a number of standard procedures for assessing the susceptibility of high strength steel to hydrogen-assisted cracking (9–21), none of these methods address the effect of electrochemical potential and pH, both of which are real characteristics of the environment where these steels are used.

Determination of the optimum pH and electrochemical potential where a material is most tolerant to hydrogen can be achieved in several ways. One way is to measure the critical stress intensity, K_{Ic} , at various artificially generated electrochemical potentials and over a range of pH. Although artificially created, the resulting data could be referred to as the critical stress intensity for environmentally assisted cracking (K_{IEAC}) with the understanding that equilibrium conditions in the environment will be established with the coated substrate; these conditions include the parameters expressed in equation 1. The equilibrium potential for the hydrogen proton reaction in water is as follows:

$$E_{H^+/H_2} = -0.059 pH - \left(\frac{0.059}{2} \right) \log \rho_{H_2} \quad [1]$$

The potential varies as a function of pH and hydrogen pressure. Therefore, the likelihood to produce hydrogen gas or monatomic hydrogen increases as electrochemical potential and pH decrease. Strong effects of these parameters on the measured critical stress intensity have been demonstrated (8).

2. Experimental Procedure

It was concluded, based on numerous conversations with the Environmental Security Technology Certification Program, that the most frequently used high strength steels in critical military applications were vacuum arc re-melted (VAR) 4340 at various strength levels, PH¹ 13-8 molybdenum steel in the H1000 condition, and 4335V steel. The VAR material was tested in the Rockwell C hardness (HRC) 38 and HRC 52 conditions. The VAR 4340 had two heat treatments: a) an 845° C austenitizing treatment for 30 minutes followed by an oil quench and a 340° C, 1-hour temper which yielded a hardness of 52 HRC, and b) an 845° C austenitizing treatment for 30 minutes followed by an oil quench and a 470° C temper for 1 hour which yielded a hardness of 38 HRC. The supplied chemical composition and mechanical properties are presented in tables 1 and 2.

Table 1. Chemical composition of supplied experimental materials.

Element	VAR 4340	PH 13-8 Molybdenum	4335
Carbon	0.42	0.032	0.36
Manganese	0.77	0.03	0.72
Silicon	0.28	0.05	0.28
Phosphorus	0.009	0.007	0.008
Sulfur	0.002	0.0004	0.009
Chromium	0.76	12.45	0.75
Nickel	1.76	8.25	1.83
Molybdenum	0.23	2.20	0.37
Cobalt	NA	0.02	NA
Aluminum	NA	1.03	NA
Nitrogen	NA	0.0030	NA
Titanium	NA	0.018	NA
Vanadium	NA	0.02	0.20
Oxygen	NA	0.0002	NA
Iron	Remainder	Remainder	Remainder

¹precipitation hardenable

Table 2. Mechanical properties of supplied experimental materials.

Mechanical Property	VAR 4340 HRC 38	VAR 4340 HRC 52	PH 13-8 Molybdenum	4335
Yield strength, MPa	1070	1550	1460	1450
Tensile strength, MPa	1200	1875	1520	1655
Elongation, percent	16	-	15	10
Reduction of area, percent	-	-	63	-
Hardness, HRC	38	52	46	-
Plain strain fracture toughness	105 MPa√m	60 MPa√m	100 MPa√m	100 MPa√m

This work used pre-cracked charpy impact specimens in an 8- to 10-hour slow strain, constant displacement rate (0.005 mm/min), double cantilever beam experiment on an Instron Model 4301 testing apparatus to determine K_{IEAC} . Schematics of the test specimen and testing apparatus are presented in figures 1 and 2, respectively. The test specimens were manufactured in accordance with ASTM-E-399 (21). The pre-cracking took place in three separate stages that incorporated decreasing loading levels. In the initial pre-cracking stage, the load was maintained to keep stress intensity values below 80% of the estimated experimental critical stress intensity, and the stress ratio ($\sigma_{max}/\sigma_{min}$) was kept between -1 and $+0.1$. During the final stage of pre-cracking, the load was reduced so the final value of K_{max} was unlikely to exceed 60% of the estimated value for K_I during experimentation. Additionally, the final value for K_{max}/E did not exceed $0.0032 \text{ m}^{1/2}$, in which E is Young's Modulus. Pre-crack length, represented by the dimensionless expression of crack length over specimen width (a/W), was maintained near 0.5.

The calculation for the onset of environmentally assisted cracking, or K_{IEAC} , was derived as follows for cantilever bending from the four-point bending expression:

$$K_{IEAC} = \left(\frac{6M_{IEAC} \sqrt{\pi a}}{BW^2} \right) xf(a/W) \quad [2]$$

in which

$$f(a/W) = 1.122 - 1.40(a/W) + (7.33(a/W)^2 - 13.08(a/W)^3) + 14.00(a/W)^4 \quad [3]$$

in which

M_{IEAC} = the applied moment necessary to induce cracking,

B = the specimen thickness,

W = the specimen depth, and

a = the initial crack length.

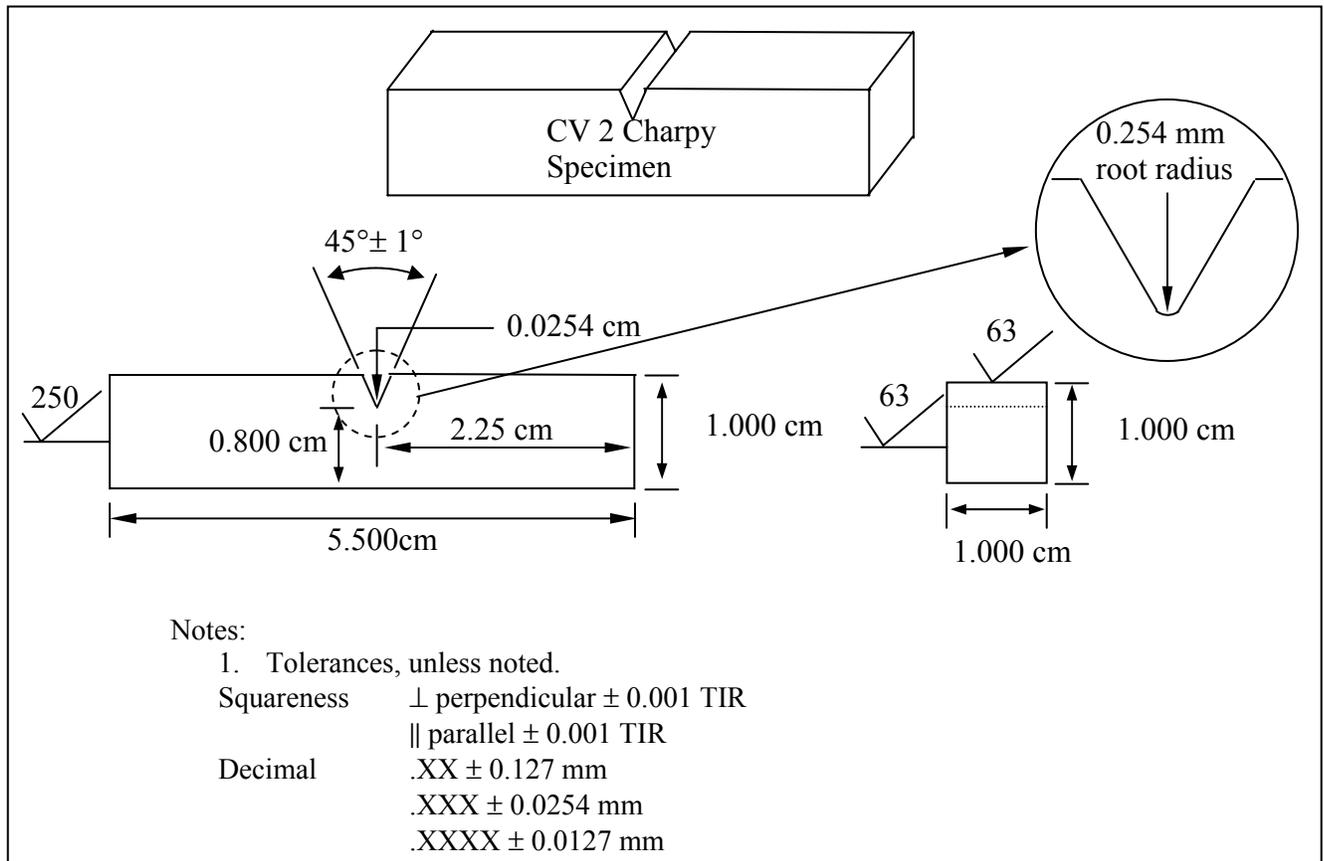


Figure 1. Schematic of the K_{IEAC} fracture specimen.

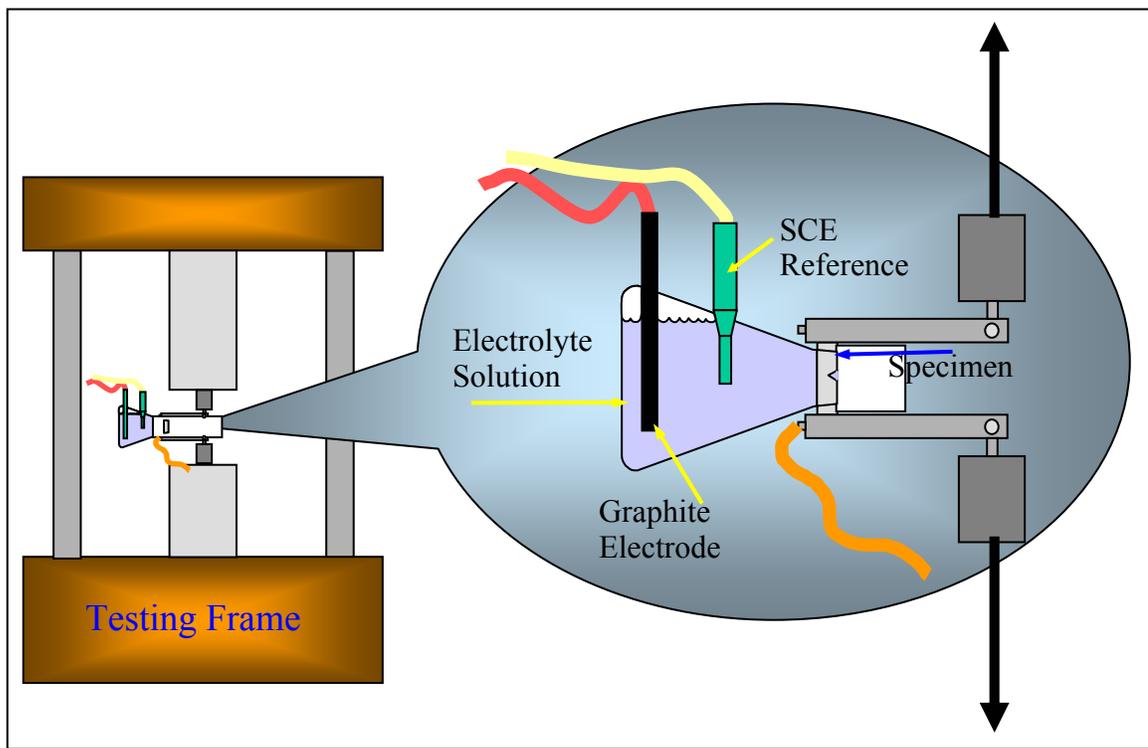


Figure 2. Schematic of the K_{IEAC} fracture apparatus.

For double cantilever bending, the term M_{IEAC} becomes $P_{IEAC}S$, in which P_{IEAC} is the load necessary to induce critical cracking and S is the length of the moment arm. This yields the used expression for K_{IEAC} .

$$K_{IEAC} = \left(\frac{6P_{IEAC}S\sqrt{\pi a}}{BW^{3/2}} \right) xf(a/W) \quad [4]$$

in which

$f(a/W)$ varies between 2.3 and 3.2 for a/W values between 0.45 and 0.55, respectively.

Overall crack length was determined from optical fractographs of each specimen. The final crack length was determined from the average of the summation of length measurements perpendicular to the crack front. Computer software digitally analyzed the data. First, the photo was digitized, and then the software outlined the edges of the notched and pre-cracked region. Next, the software filled the notched and pre-cracked area, and subsequently calculated lengths across this filled area. The lengths were then averaged. This method is depicted in figure 3 with a step size of 20 shown for clarity. The method used had a step size of 1 for maximum accuracy.

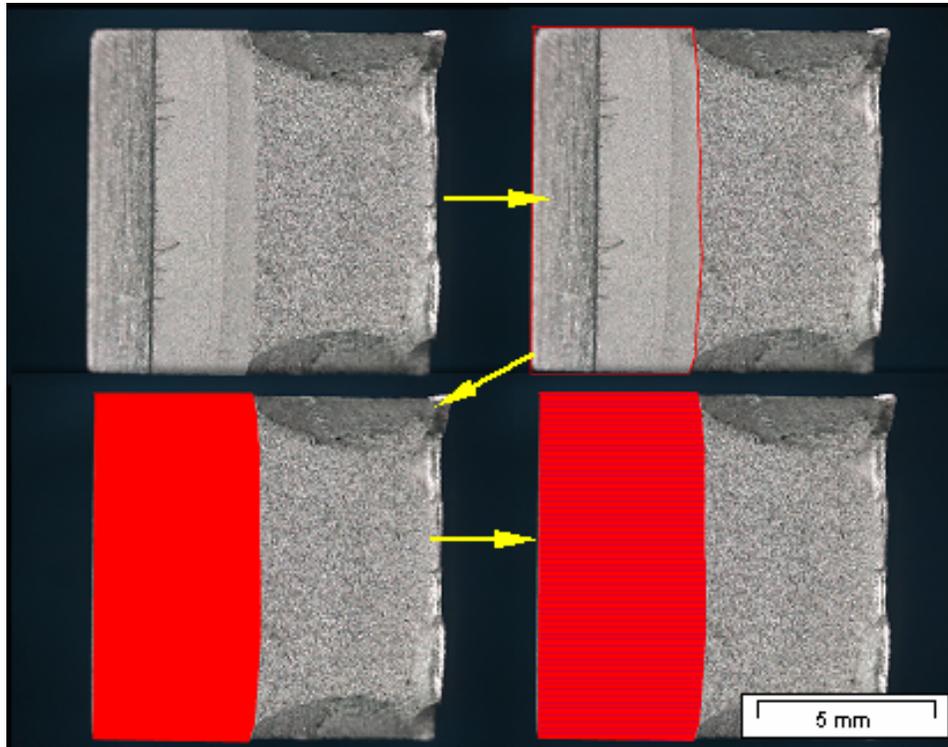


Figure 3. Crack length determination procedure.

Experimentation was conducted in a range of electrochemical potentials and pH. Each material was subjected to neutral 3.5% sodium chloride (NaCl) solution at four different electrochemical potentials: -0.6 , -0.8 , -1.0 , and -1.2 volts saturated calomel electrode (V_{SCE}). Electrochemical

potential was maintained with the use of an Aeromedical Equipment Laboratory Instruments Model 2051 potentiostat and a potassium chloride reference. Tests were conducted in dilute sulfuric acid at pH 2, and in dilute sodium hydroxide at pH 11, at both -0.6 and -0.8 V_{SCE} . Open circuit experiments were also conducted in neutral 3.5% NaCl. Five specimens were tested for each group.

3. Results

Figure 4 presents the results from the VAR 4340 steel heat treated to HRC 52. These results were obtained in the 3.5% NaCl, in the diluted sulfuric acid, and in the sodium hydroxide solutions. This material showed the least dependence on electrochemical potential and on pH of all the materials investigated. The measured K_{IEAC} values were greatest (22 $MPa\sqrt{m}$ in the NaCl and pH 11 solutions at -0.6 V_{SCE}), while the lowest measured K_{IEAC} values, 16 $MPa\sqrt{m}$, were obtained from the NaCl solution at -1.2 and -1.0 V_{SCE} . The measured open circuit K_{IEAC} value in 3.5% NaCl was approximately 17.5 $MPa\sqrt{m}$ at the measured open circuit potential of -0.57 V_{SCE} . The caustic pH levels slightly increased the measured K_{IEAC} values at -0.8 V_{SCE} , although they did not appear to significantly increase them at -0.6 V_{SCE} versus those obtained in 3.5% NaCl solution at the same potential. Additionally, the acidic solution generally decreased the measured value of K_{IEAC} at both -0.8 and -0.6 V_{SCE} , versus those obtained in 3.5% NaCl solution at the same potentials.

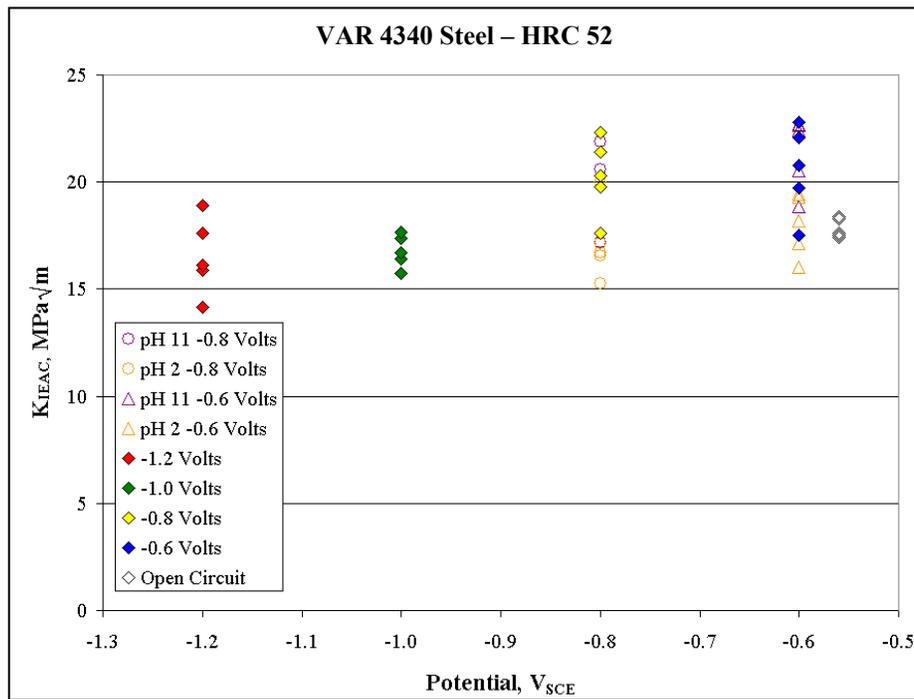


Figure 4. K_{IEAC} results for VAR 4340 HRC 52.

The results obtained from VAR 4340 steel heat treated to HRC 38 are presented in figure 5. The 3.5% NaCl, pH 2 and pH 11 solutions were used. The material demonstrated a great dependence on both potential of hydrogen and on electrochemical potential. The greatest measured K_{IEAC} value, 90 MPa \sqrt{m} , was obtained at $-0.8 V_{SCE}$ in the pH 11 solution. The lowest measured K_{IEAC} value, 18 MPa \sqrt{m} , was obtained at $-1.2 V_{SCE}$ in the NaCl solution. The pH 11 solution tended to increase the measured K_{IEAC} values considerably at $-0.6 V_{SCE}$, versus those obtained in 3.5% NaCl solution at the same potential. Additionally, the acidic solution decreased the measured value of K_{IEAC} significantly at $-0.8 V_{SCE}$, while the measured K_{IEAC} values remained largely unaffected at $-0.6 V_{SCE}$ versus those obtained in 3.5% NaCl solution at the same potentials. The measured open circuit K_{IEAC} value in 3.5% NaCl was approximately 65 MPa \sqrt{m} at the measured open circuit potential of $-0.59 V_{SCE}$.

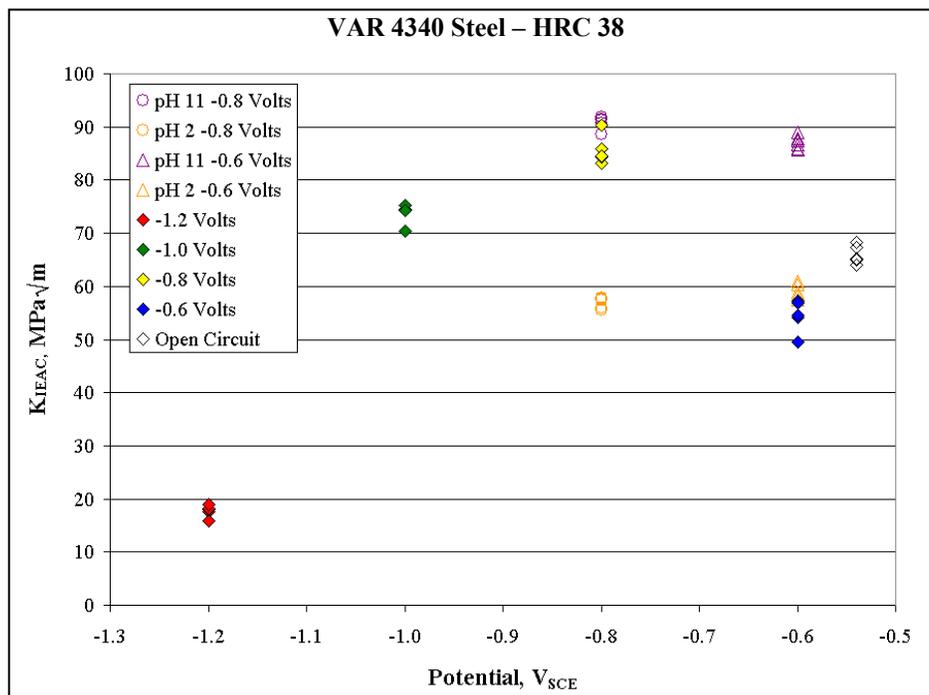


Figure 5. K_{IEAC} results for VAR 4340 HRC 38.

The results from the PH 13-8 Mo in the H1000 condition are presented in figure 6. Again, K_{IEAC} values were obtained from all three solutions. The highest measured K_{IEAC} values were 78 MPa \sqrt{m} and were obtained from the pH 11 solution at $-0.8 V_{SCE}$. The lowest measured K_{IEAC} value, 9 MPa \sqrt{m} , was obtained at $-1.2 V_{SCE}$ in the NaCl solution. The measured open circuit potential of $-0.76 V_{SCE}$ yielded a K_{IEAC} value in 3.5% NaCl of 72 MPa \sqrt{m} . Similar to the other materials, the pH 11 solution raised the measured K_{IEAC} values while the pH 2 solutions lowered the measured values.

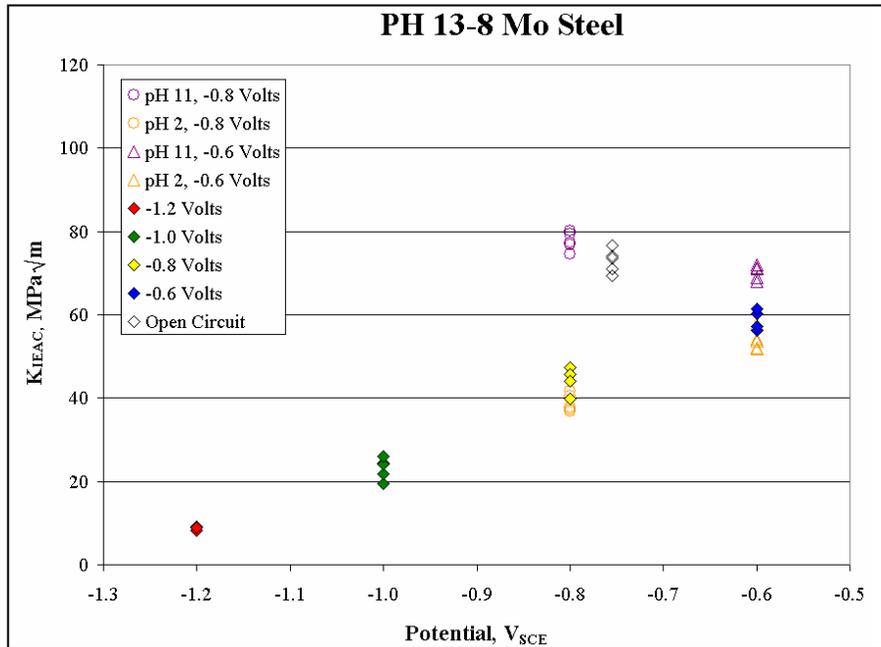


Figure 6. K_{IEAC} results for PH 13-8 molybdenum.

The 4335 material exhibited the same general trend as the other materials. The results for this material are presented in figure 7. The highest measured value for K_{IEAC} , 84 $MPa\sqrt{m}$, was obtained in the pH 11 solution at $-0.8 V_{SCE}$. The lowest measured value was again at $-1.2 V_{SCE}$ and was approximately 22 $MPa\sqrt{m}$ in 3.5% NaCl. The measured open circuit K_{IEAC} value was 67 $MPa\sqrt{m}$ at the measured open circuit potential of $-0.59 V_{SCE}$.

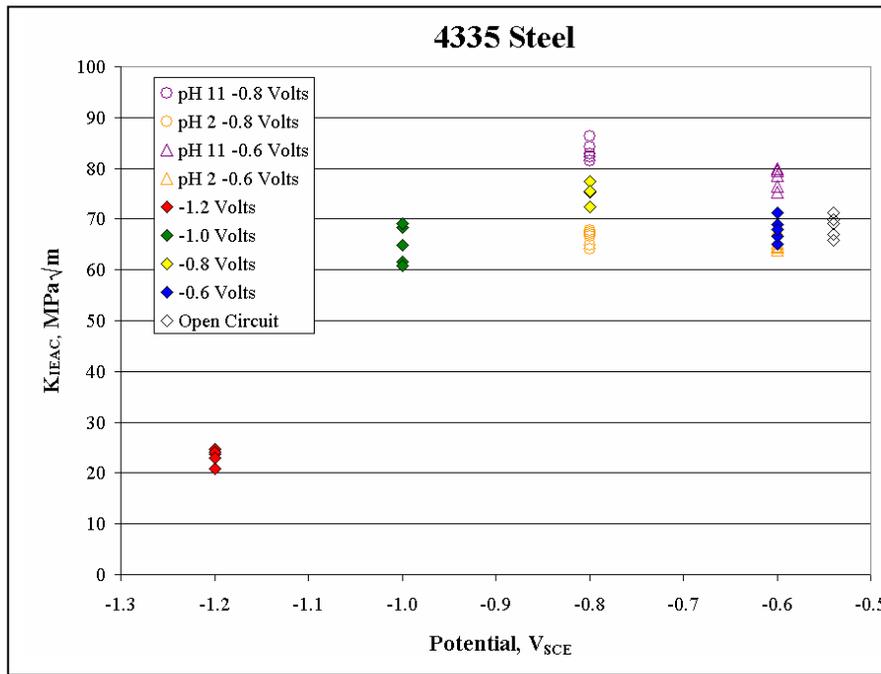


Figure 7. K_{IEAC} results for 4335 steel.

The surfaces of the fractured specimens were characterized for fracture morphology within the different regions. Each of the three regions (cyclic fatigue transgranular fracture, hydrogen-affected brittle intergranular or semi-intergranular fracture, and semi-ductile or ductile fracture) was observed in the materials investigated. Figures 8 through 11 depict the three zones of fracture for VAR 4340 HRC 52, VAR 4340 HRC 38, PH 13-8 molybdenum, and 4335 steel, respectively.

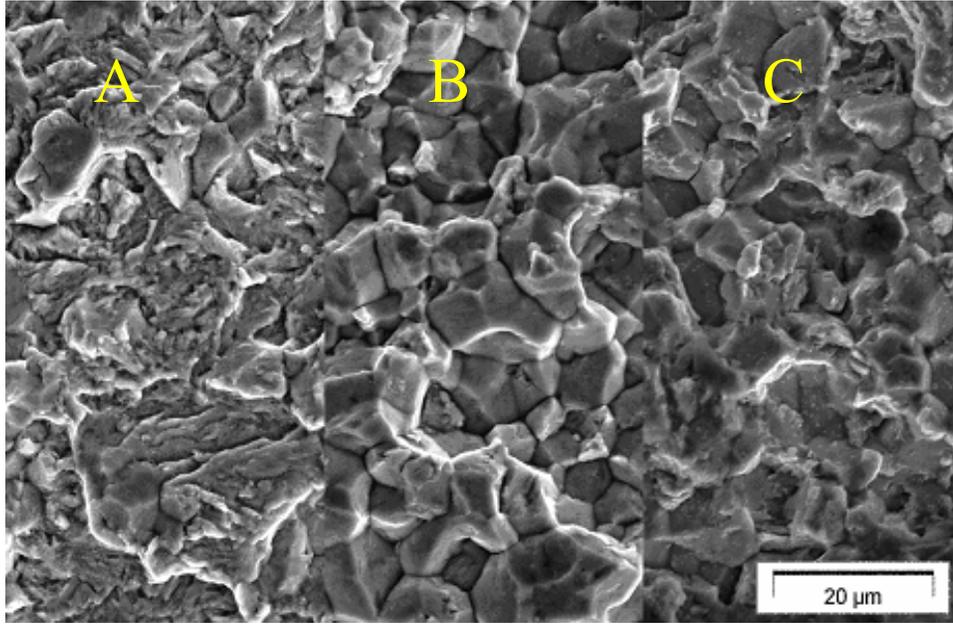


Figure 8. Fracture morphology zones for VAR 4340 HRC 52 steel.

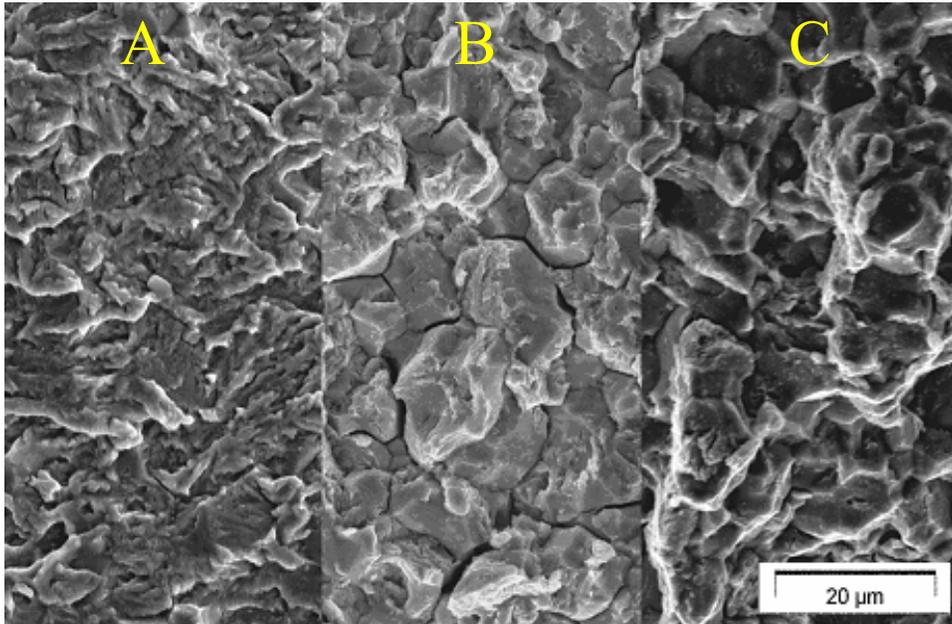


Figure 9. Fracture morphology zones for VAR 4340 HRC 38 steel.

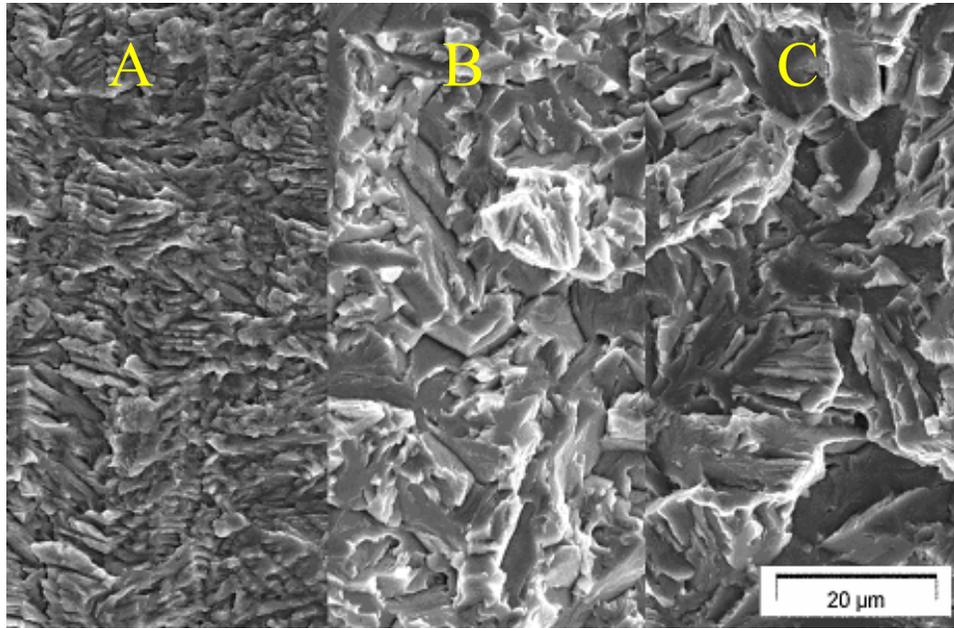


Figure 10. Fracture morphology zones for PH 13-8 molybdenum steel.

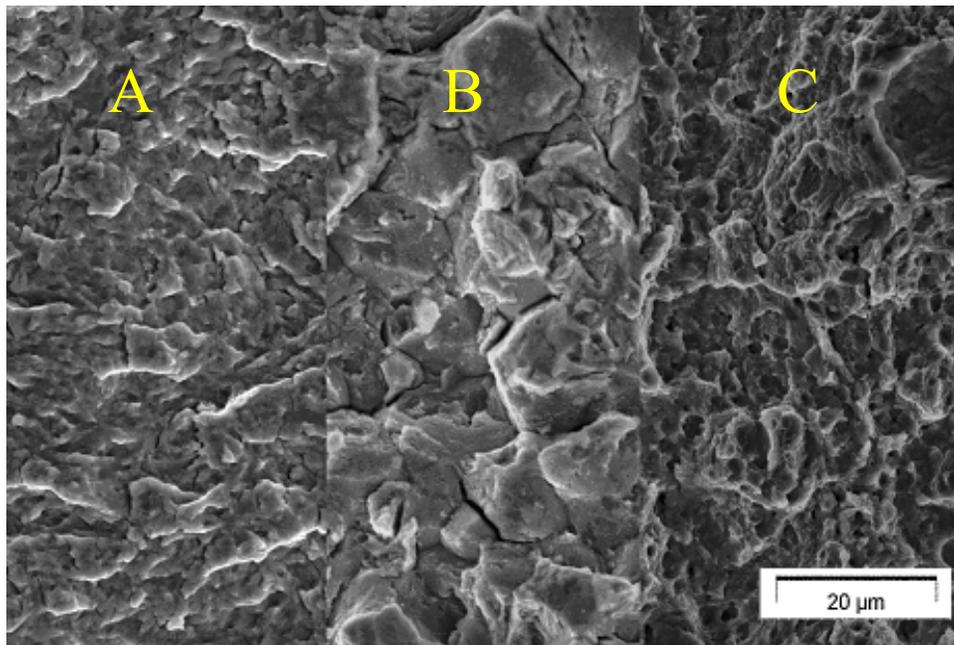


Figure 11. Fracture morphology zones for 4335 steel.

4. Discussion

The materials demonstrated strong dependence with electrochemical potential. Additionally, the general trend of the pH 11 solution to increase the measured K_{IEAC} values while the pH 2 solution decreased the measured values held true for all materials. It appeared that the initial relationship governing the dissociation of the hydrogen proton reaction dominated the K_{IEAC} experimentation. It was interesting to note that the PH 13-8 had a nearly direct relationship with potential; as potential decreased, so did the measured K_{IEAC} value. VAR 4340 in the HRC 52 condition remained largely unaffected by electrochemical potential and pH, probably because of the sensitivity of the material to hydrogen and the fact that general corrosion of 4340 in 3.5% NaCl was sufficient to introduce enough hydrogen in the solution to deleteriously affect the steel. The fracture toughness of the materials decreased dramatically in the presence of excess hydrogen. The K_{IEAC}/K_{Ic} ratios for the tested materials are presented in table 3.

Table 3. K_{IEAC}/K_{Ic} ratios for the tested materials.

Material	PS K_{Ic} (MPa\sqrt{m})	K_{IEAC} (MPa\sqrt{m}) at -1.2 V_{SCE}	$K_{IEAC}/PS K_{Ic}$
VAR 4340 HRC 52	60	17	0.28
VAR 4340 HRC 38	105	17	0.16
PH 13-8 Mo	100	9	0.09
4335	100	23	0.22

Since most of these materials are used in conjunction with sacrificial coatings because of their poor corrosion resistance, designers must be careful to account for hydrogen damage when attempting to avoid critical flaws. This case is more extreme for PH 13-8 molybdenum, for example, since its K_{IEAC}/K_{Ic} ratio is 0.09. A designer would be more likely to allow a critical flaw in this material, when ignoring hydrogen, than in VAR 4340 HRC 52. The K_{IEAC} values in the presence of excess hydrogen are actually very similar. Realistically, this is an exaggeration because of the extreme amount of hydrogen at $-1.2 V_{SCE}$, but the trend can certainly be seen, even at other potentials, in the plotted data. The minimum and maximum measured K_{IEAC} data are summarized in Table 4. Coating system designers can use these data in their designs. By tailoring the coating to push the overall potential of the coated substrate toward $-0.8 V_{SCE}$ and toward the basic end of the potential of hydrogen scale, we would have a design that yields the most hydrogen-tolerant system possible for each material, although the specific values vary slightly from case to case.

Table 4. Maximum and minimum measured K_{IEAC} values for the tested materials.

Material	PS K_{Ic} (MPa√m)	Maximum K_{IEAC} (MPa√m), Potential and pH at Maximum	Minimum K_{IEAC} (MPa√m), Potential and pH at Minimum
VAR 4340 HRC 52	60	21.4, -0.6 V_{SCE} , pH 11	16.5, -1.2 V_{SCE} , neutral 3.5% NaCl
VAR 4340 HRC 38	105	91.2, -0.8 V_{SCE} , pH 11	17.4, -1.2 V_{SCE} , neutral 3.5% NaCl
PH 13-8 Mo	100	78.3, -0.8 V_{SCE} , pH 11	9.1, -1.2 V_{SCE} , neutral 3.5% NaCl
4335	100	84.2, -0.8 V_{SCE} , pH 11	23.4, -1.2 V_{SCE} , neutral 3.5% NaCl

5. Conclusions

The detrimental effect of hydrogen on K_{Ic} was characterized over a wide electrochemical potential and pH range for the high strength steels investigated in this work. Most of the materials demonstrated extreme dependence of K_{IEAC} on both electrochemical potential and potential of hydrogen. PH 13-8 molybdenum proved to be the most affected. The K_{IEAC} value was lowered to approximately 10% of the known plane strain fracture toughness, K_{Ic} , value in the presence of hydrogen. The other materials behaved similarly but were not as severe. The maximum K_{IEAC} values for the materials investigated in neutral 3.5% NaCl were as follow: VAR 4340 HRC 52, 21 MPa√m; VAR 4340 HRC 38, 86 MPa√m; PH 13-8 molybdenum, 59 MPa√m; and 4335 steel, 75 MPa√m.

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US ARMY RSCH LABORATORY
ATTN AMSRL CI OK TECH LIB
2800 POWDER MILL RD
ADELPHI MD 20783-1197

1 DIRECTOR
US ARMY RSCH LABORATORY
ATTN AMSRL D D SMITH
2800 POWDER MILL RD
ADELPHI MD 20783-1197

ABERDEEN PROVING GROUND

2 DIRECTOR
US ARMY RSCH LABORATORY
ATTN AMSRL CI OK (TECH LIB)
BLDG 305 APG AA

20 DIRECTOR
US ARMY RSCH LABORATORY
ATTN AMSRL WM MD S GRENDAHL
BLDG 4600

4 DIRECTOR
US ARMY RSCH LABORATORY
ATTN AMSRL WM MC C MILLER
B PLACZANKIS J KELLEY
R ADLER
BLDG 4600