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Control of Stress Corrosion Cracking by Inhibitors
(A Review of the Literature)

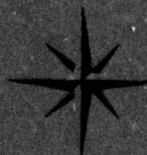


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CONTROL OF STRESS CORROSION CRACKING BY INHIBITORS

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ABSTRACT

Studies of inhibitors of stress corrosion cracking (SCC) are reviewed for austenitic stainless steels, brass, titanium, aluminum alloys, and carbon and low alloy steels. There are at least five combinations of alloy and environment for which inhibitors have provided engineering solutions for the control of SCC, and others are not improbable at an early date. In view of the complexity of the problem and the comparatively few sustained research programs in this special field, it is not surprising that the fundamentals of inhibition of SCC are incompletely understood.

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I. INTRODUCTION

Stress corrosion cracking (SCC) is a process involving the nature of an alloy (metallurgy), stress (mechanics), and environment (chemistry or electrochemistry). All possible countermeasures to SCC may be assigned to these three categories, metallurgy, mechanics and electrochemistry (including temperature), and in engineering practice measures from any or all three categories may be employed. One may for example select cupronickel condenser tubing to replace admiralty metal tubing to avoid ammoniacal SCC, representing a metallurgical approach to the problem. Or one may employ a thorough stress-relieving anneal of an assembled admiralty metal heat exchanger, representing a mechanics approach. And one may elect to apply cathodic protection, as by spraying stainless steel with lead to prevent chloride SCC, representing an electrochemical approach.

Changing the composition of the environment can also be regarded as a chemical or electrochemical approach, usually pursued by identifying a contaminant species responsible for SCC (such as chloride in austenitic stainless steel systems) and lowering the concentration of this species below what is hopefully a safe threshold value.

Another possible approach to controlling SCC by controlling the environment is by the use of additives. These additives may act in three distinct ways: (1) They may destroy or prevent the formation of the cracking species, as by the use of soda ash or ammonia to prevent polythionic acids cracking of sensitized stainless steel. (2) They may move the potential of the alloy into a safe range. Or, (3) they may enter directly into blocking of one or more of the chemical reactions responsible for SCC. This present survey of these additives, or inhibitors, will summarize the work to date regardless of which of the three categories of mechanisms above is operative, partly because in many instances the mechanism is unknown or only suggested. Although the present conference is concerned largely with coatings which are not appropriate to heat exchanger technology, the inhibitor work on heat exchanger materials is included on the chance that some of the principles involved may be useful in formulating thoughts about inhibitors in coatings.

The material is organized according to alloy family.

II. AUSTENITIC STAINLESS STEELS

These alloys undergo SCC in warm or hot chloride solutions, in hot caustic solutions, in strong acids containing chloride at

room temperature, and (if sensitized) in polythionic acids at room temperature.

In hot chloride solutions Staehle, Beck and Fontana showed that reducing adventitious oxygen by reacting with hydrogen eliminated SCC (1), perhaps by depressing the potential into a protective range. Other studies, by Williams and Eckel (2), Rion (3), Edeleanu and Snowden (4), and Phillips and Singley (5) showed that deoxygenation by hydrazine was a countermeasure to SCC, again possibly by depressing the potential. Sulfite was found to be a somewhat less effective inhibitor (2, 4, 5). Although this deoxygenation approach is not considered to belong to the strictly inhibitor literature, it is noted here as it suggests that inhibitors may be sought for some alloys by consideration of the effects of additives on potential. Actually this approach (depressing the potential) was suggested by much earlier work by Mears, Brown and Dix who demonstrated the efficacy of cathodic protection against chloride SCC in stainless steel, with potential data (6). H. van Droffelaar has shown that sprayed coatings of metallic lead will prevent chloride SCC of stainless steel (7); O'Dell and Sarada have shown that this effect is due to cathodic protection and not to classic inhibition by lead species (8).

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Under various conditions, chromate may either be indicated to counter SCC (9, 10, 11, 12), or it may be counterindicated (4, 13). Perhaps this variability in response may reflect a competition between the beneficial effect of tending to stabilize a protective film versus the adverse effect of raising the potential into a hazardous range. Alkali phosphates have been reported to inhibit to various degrees chloride SCC of steel if unsensitized (2, 4, 10, 11, 12, 14), but perhaps the ratio of inhibitor to chloride is an important factor (11, 13).

Snowden found nitrite an effective inhibitor providing the molar ratio $\text{NaCl}/\text{NaNO}_2$ was 1/1, but not if the ratio was 2/1 (13), and Couper found it only slightly inhibiting at a much higher $\text{Cl}^-/\text{NO}_2^-$ ratio, in 42% MgCl_2 (12).

Couper (12) and Uhlig and Cook (15) reported prevention of SCC in boiling 42% MgCl_2 with 2% NaNO_3 . Fujii and Kumada reported work in 35% MgCl_2 showing that the inhibiting character of various polyoxyanions decreases as follows: $\text{NO}_3^- > \text{H}_2\text{PO}_4^- > \text{CrO}_4^{2-} = \text{WO}_4^{2-}$, from which they concluded that there is no relationship between oxidizing power and inhibition (16). Uhlig and Cook showed that the action of the nitrate is to shift the protection potential (or "critical cracking potential") with respect to the corrosion potential to put the corrosion potential in the protected range (15). The efficacy of nitrate is judged to be sufficient for it to be called out as a preventive measure in NACE Recommended Practice RP-01-70 (17). If the nitrate is not present in sufficient concentration, SCC will occur, as shown by

Uhlig and Cook (15) and as may be inferred from the work of Edeleanu and Snowden (4).

Uhlig and Cook also found iodide and benzoate to be inhibitive in 42% $MgCl_2$ (15).

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Holzworth and Symonds (18) found that dipping the steel in lithium silicate inhibited SCC in dilute acidified chloride solution at 90 C. Bergen found that sodium metasilicate somewhat inhibited SCC in $CaCl_2$ at 132 C, and burnishing the steel with silver inhibited SCC in short-time tests in that environment (19).

Overman used radioactive tracers to study inhibition of SCC in Type 304 stainless steel in boiling solutions of 10 - 100 ppm chloride plus the selected substances. One to 50 ppm silicate, phosphate, nickelous, iodide, bromide or chromate ions inhibited crack formation in the standard solution. The tracer studies revealed that corrosion and crack formation were inhibited by cations (complexes?) or anions that compete with the chloride ions for the available corrosion sites on the surface of the steel (20).

Podobaev and his associates studied the inhibition of SCC in $MgCl_2$ boiling at 153 C. KI acted as a partial inhibitor by itself; when combined with BA-12 (a condensation product of benzylamine and paraform-aldehyde) there is a synergistic action to effect complete protection against SCC. Another inhibitor designated PB-5) (not further described) also protected completely against SCC (21). Katapin, a surface-active substance used as a pickling restrainer, retards the anodic process by forming a protective film, but it is incompletely effective in inhibiting SCC (22).

Podobaev showed that $SnCl_2$ added to boiling $MgCl_2$ (153 C) produced a large increase in survival time of stainless steel; there was some inhibition from additions of $SnCl_4$, $PbCl_2$, $SbCl_3$, and $BiCl_3$. The addition of $CdCl_2$ at 45 millimolar concentration produced a 5-fold increase in survival time. Podobaev explained the results on the basis of changing cathodic polarization characteristics (23). Beauchamp showed that if the chloride were present as 1705 ppm of $CdCl_2$ at 205 C, 18-8 stainless steel did not crack under a stress of 10 ksi in 8 hours, but if stressed at 26.5 ksi, SCC occurred in 17 hours (24). Leu and Helle found that the addition of $SnCl_2$, metallic magnesium or metallic zinc to boiling $MgCl_2$ prevented or spectacularly reduced SCC in type 304 stainless steel (25). O'Dell's study of sensitized type 304 stainless steel in 4 N NaCl with pH 2.3 at 100 C found that the additives NaI, $SnCl_2$, $SnCl_4$, $FeCl_3$, $SnSO_4$, $CdSO_4$, CdI and n-butylamine prevented SCC of U-bend specimens for 600 hours as compared with 24 hours required for SCC in the uninhibited system. All of these substances permitted pitting and/or general corrosion except for $CdSO_4$. Cd^{++} and SO_4^{--} were only moderately effective

when added separately; however, CdSO_4 produced a synergistic effect preventing all forms of corrosion. Polarization data showed that the inhibitor has a pronounced effect on the anodic reaction. EDAX analysis of the material within cracks initiated in uninhibited solution, to which CdSO_4 was then added, showed high concentration of the inhibitor. The inhibition was attributed to the synergistic effects of the cadmium polymerizing with the chloride combined with a desorbing action of the sulfate ion upon the chloride (26).

Although chloride is clearly a causative agent for SCC in stainless steel, the cation apparently has a significant role in the corrosion process. Varying the cation in chloride solutions can produce changes ranging from acceleration of SCC to blockage of the corrosion process (27, 28, 29). THIS PAGE IS BEST QUALITY PRACTICABLE FROM COPY FURNISHED TO DDG

Several groups have studied the aggressive behavior of metal chloride solutions with varying results (29-33). Perhaps the best order of ranking for four cations is as follows: Li^+ (most aggressive), Mg^{+2} , Ca^{+2} , Na^+ (34).

Zucchi and associates have reported on a series of researches on inhibition of SCC of austenitic stainless steels in strong acid chloride solutions at room temperature, using various organic inhibitors (35-40). Benzonitrile completely inhibited transgranular SCC in type 304 steel probably by interfering with the anodic reaction, and benzotriazole decreased SCC attack; the environment was 0.5 N NaCl acidified with H_2SO_4 (35). In 2 N HCl, phenyl thiourea completely blocked attack, again by blocking the anodic reaction; di-n-butyl thiourea also blocked all attack, as did the combination of KI + n-decylamine (36). In 2 N HCl, di-o-tolyl thiourea; benzoimidazole-2-thiol; N-N'-diphenyl thiourea; sulfadiazole-3-thiol; and benzothiazole-2-thiol completely blocked "fissuring" (cracking/) (37-39). Again in 2 N HCl but this time with type 316 steel the inhibition of SCC by benzimidazole-2-thiol; benzothiazole-2-thiol; sulfathiazole-3-thiol; and N-N'-diphenyl thiourea is due to blocking the anodic reaction. All the organic substances examined altered the hydrogen reaction, but only those substances which inhibited the anodic reaction prevented SCC. The proposed mechanism is one of rapid adsorption onto the metal exposed by stress to block the anodic reaction. There does not seem to be any correlation between inhibiting general corrosion and inhibiting fissuring (40).

Lafranconi et al. have studied SCC in austenitic stainless steel also in strongly acidic chloride media at room temperature, and they found inhibition by traces of Pb^{+2} . This inhibition they showed was due to effects on the cathodic reaction (hydrogen reduction), which they attributed to the effect of lead on hydrogen overvoltage (41). Perhaps the apparent discrepancy between this finding and that of O'Dell and Sarada (8) can be assigned to a difference in cracking mechanisms, as the latter were working with (only slightly acidic) MgCl_2 at 153 C.

One inhibitor of SCC in stainless steel in a strong acid--white fuming nitric acid--which found its way into engineering practice but not into the technical literature is 1% HF (42).

III. BRASS

Gisser found that continuous resin films such as phenol formaldehyde delayed attack on brass in ammonia (43). Whether there is any intrinsic inhibiting effect of substances in the film or whether only barrier properties are involved does not seem to be established. Rowlands protected aluminum brass (C68700) against SCC in sulfur-polluted seawater with sodium dimethyl-dithiocarbamate which forms a protective copper-chelate film on the brass surface (44). Poly-hexamethylphosphate and sodium chromate have also been found to prevent stress corrosion cracking of aluminum brass (C68700) in recirculating seawater but not in fresh water (45). Uhlig and his students found that with 63-37 brass exposed to ammoniacal and to tartrate solutions, delay in cracking occurred with the addition of bromide ions and also chloride ions. The delay was attributed to the shift of the protection potential (or critical cracking potential) in the positive direction with respect to the corrosion potential (46). Podobaeu and his associates have worked on the problem of protecting cartridge brass against corrosion and SCC by protective coatings of greases and oils, with and without inhibitive additions. Though not perfectly protective, potassium ferrocyanide and dibutyl phthalate have inhibitive characteristics attributed to the formation of protective complex reaction products (47). Walston attributed the SCC of an admiralty heat exchanger exposed to ammonium chloride to ammonia released by reaction of the chloride with the brass, combined with the absence of hydrogen sulfide which would have acted as an inhibitor (48).

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IV. TITANIUM

Kiefer and Harple showed that in red fuming nitric acid 1% NaBr could inhibit SCC in commercially pure titanium (75A) at room temperature (49). Rittenhouse found that the addition of water to red fuming nitric acid would control SCC in pure titanium and in the Ti-8% Mn alloy (50). A Military Specification has been written calling for up to 14% NO₂ and 2.5% H₂O, balance HNO₃.

Haney and Wearmouth showed that additions of water inhibited SCC initiation in titanium in methanol (51). This inhibition is not effective in the presence of a crack or of chloride.

Pressurized fuel tanks of Ti-6Al-4V experience SCC if they contain N_2O_4 of sufficiently high purity, but the addition of NO inhibits the SCC (52). This inhibition is sufficiently practical as a solution to the SCC problem that a NASA Specification (PPD-2B) calling for 0.60 - 1 percent NO was written and has been successfully applied in the U. S. space program.

The addition of oxygen gas has been found to inhibit cracking of titanium tanks containing gaseous hydrogen (53).

The inhibition of SCC in titanium alloys is poorly understood, and little has been suggested concerning the mechanism(s).

V. ALUMINUM

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Protection of high strength aluminum alloys from SCC in moist atmospheres is promoted by a primer containing the inhibitor strontium chromate (MIL-P-23377), widely used in aerospace weapons systems. Zinc chromate primer confers some considerable protection for atmospheric service, and even more so when further covered by an epoxy paint system (54).

Galvanic coatings are practical solutions to the SCC problem for high strength aluminum alloys. Zinc-rich epoxy, zinc electroplate, and metallized 7072 coatings have been shown to greatly extend the life of 2014 and 7079 alloys (54). Magnesium pigment added to inorganic binders (55) or epoxy primer (56) has given good results.

A corrosion prevention compound has been developed by personnel of the Naval Air Development Center which contains two unidentified inhibitors (57). Although only corrosion tests, not stress corrosion tests, have been reported to date, the results of the corrosion tests are sufficiently impressive to suggest that tests of the compound as a countermeasure to SCC would be interesting. The compound, named AMLGUARD, is covered by military specification MIL-C-85054(AS).

VI. CARBON STEELS AND LOW ALLOY STEELS

Caustic cracking of boiler plate was once a serious problem for which inhibitor technology contributed many countermeasures. Straub lists phosphate, tannate, chromate, nitrate, acetate, and arsenic as inhibitors of this failure mode. He also showed that a ratio of sulfate to alkalinity of 2.1 would control cracking (58). Schroeder and Berk found that in high pressure boilers the addition of nitrates to 40% of the alkalinity inhibited

cracking, and sodium sulfate was also effective. Quebracho inhibited cracking by removing oxygen and by keeping the calcium phosphate and calcium carbonate dispersed. The addition of waste sulfite liquor to the water in locomotive boilers inhibited cracking (59). Several inhibitors of cracking in boiler plate were discussed in a symposium on caustic embrittlement. These additives included R_2O_3 (where $R=Fe$ and Al) (20), organic extracts (60), nitrate (61-63), phosphate (61), waste sulfite liquor (62) (for oxygen removal), quebracho (63), myrabalans (63), and combinations of sodium nitrate and lignin sulfonate (63) and of sodium nitrate and tannin (64). No mechanism of inhibition was proposed at this symposium. Extensive use has been made of coordinated phosphate and pH treatment to protect boilers against cracking (63).

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Ammonia storage tanks of mild steel can be protected against SCC by the addition of 0.2% water (65), and this procedure is in widespread use. It is required by the Department of Transportation for transport of ammonia in quenched and tempered steel tankages (66); the technical background is given in reference 67. But condensation of vapor on cold walls of the vapor space above water-inhibited ammonia can produce a film of insufficiently inhibited ammonia, leading to SCC; the practical solution to this sort of problem is to maintain the temperature of the liquid ammonia a few degrees cooler than the walls on which condensation would otherwise occur (68). The addition of ammonium chloride or oil will also retard SCC in liquid ammonia storage tanks (69). The addition of 0.05% refrigerator oil which coats the steel surface and prevents SCC is a patented procedure (70).

Ammonium chloride will also inhibit SCC of low carbon steel exposed to nitrate solutions (71).

Dunlop was able to show that SCC of ASTM A203 Grade E steel (a low strength nickel-bearing ferritic steel) in sulfide environments was an anodic process and not one of hydrogen embrittlement; morpholine was found to be an effective inhibitor (72).

Carbonate-bicarbonate solutions have been shown capable of producing SCC in line-pipe steel, and there is considerable incentive to develop an inhibitor effective against this attack and compatible with primer materials and with cathodic protection conditions. Berry and his associates have shown that in a standard carbonate-bicarbonate solution the following substances are effective inhibitors of SCC: Sodium chromate, potassium dichromate, and zinc chromate (0.1 wt. %, based on constant strain rate tests on smooth specimens); sodium monobasic phosphate, calcium monobasic phosphate, sodium tripolyphosphate, and potassium silicate (1.0 wt. %, based on constant load tests using edge-notched flat tensile constant load tests.) Phosphate inhibitors incorporated in asphalt-based primer and coal-tar based primer were shown to be effective countermeasures to SCC in laboratory tests (73).

Anthraquinone derivatives adsorbed on AISI 4340 steel (smooth specimens) retarded SCC presumably by acting as hydrogen acceptors (74) in a system in which SCC is probably a hydrogen-assisted cracking process. NaOH, Na₂CO₃, Na₃PO₄, Na₂SiO₃, or Na₂B₄O₇ when introduced with NaNO₂ produced a synergistic inhibition of SCC in AISI 4340 steel in 10% KNO₃ at 100 C (75). Several authors have found that organic amines, including aniline and hexamethylenetetramine can inhibit SCC in high strength steels by adsorbing on the metal surface and interfering with the transport processes (76-80). Using pre-cracked specimens, Parrish et al. showed that oxidizing inhibitors could increase K_{ISCC} for a high strength steel (D6ac), but that in the presence of chloride, dichromate is not a perfect inhibitor (81). C. T. Lynch et al., working with a variety of high strength steels, were able to reduce cracking rates in most of the steels with a multifunctional inhibitor such as borax-nitrate (82). Brown, using precracked specimens, showed that the addition of enough sodium acetate to prevent the crack tip from going acid, could inhibit SCC in high strength steel (83). Johnson found that the addition of a trace of oxygen was sufficient to inhibit the growth of cracks in high strength steel under stress in gaseous hydrogen at one atmosphere pressure (84).

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VII. DISCUSSION

Considering the small number of studies there have been on inhibiting SCC compared with the vast literature on SCC in general, it is somewhat surprising that there have been so many systems (combinations of alloy and environment) in which SCC is controlled in engineering applications by the use of inhibitors: water in ammonia in steel tankages, NO in N₂O₄ in titanium tankages, water in red fuming nitric acid in titanium tankages, HF in white fuming nitric acid in stainless steel tankages, and chromate against atmospheric moisture for high strength aluminum aerospace structures. The striking thing about the inhibitor approach is that when a good inhibitor is found, it is often the only measure needed for technological success. By contrast, when other control measures are applied, they are often partial measures and are often combined with partial measures, such as various combinations of altered alloy composition, reduced strength, changed microstructure, reduced stress, and modified environment and temperature.

Stress corrosion cracking is scientifically a complex process, and it is a nucleation-and-growth process. In very few of the studies reviewed here have nucleation and growth been differentiated.

Despite long years of work by brilliant and dedicated investigators, there is hardly a single SCC system for which a

mechanism of SCC has been proposed which has met with overwhelming acceptance by most of the workers in the field. It is therefore not surprising that our knowledge about the mechanisms of inhibition of SCC is so incomplete.

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(note added in proof)

Humphries and Parkins (85) showed that NaNO_3 and KMNO_4 inhibit the cracking of annealed mild steel in NaOH by moving the potential out of the cracking range; they also found that valonea and quebracho tannin with NaH_2PO_4 prevent SCC even in the SCC potential region. These substances may interfere with the formation of an Fe_3O_4 film which may play a vital role in crack propagation in caustic (86). Berry has found eleven inhibitors of SCC in line pipe steels in (1) $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$, (2) 33% NaOH , and 20% NH_4NO_3 , as follows: CaH_2PO_4 , CaSO_4 , potassium chromate, NaH_2PO_4 , sodium orthosilicate (not effective in NaOH), sodium tetraphosphate, and tripolyphosphate (87).

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