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DMIC

REVIEW OF RECENT DEVELOPMENTS

CORROSION AND COMPATIBILITY

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CORROSION OF TITANIUM ALLOYS

Stress-Corrosion Cracking

Apollo Fuel Tank Failure

On October 25, 1966, the failure of a Ti-6Al-4V Apollo fuel tank occurred during a pressure test in which methyl alcohol procured to Federal Specification OM-232d was being used to simulate the fuel. Subsequent tests at the Manned Spacecraft Center of NASA and elsewhere have established that exposure of the heat-treated Ti-6Al-4V alloy to moderately high stresses in the presence of Federal Specification OM-232d methyl alcohol or reagent-grade methanol can lead to a form of stress-corrosion cracking failure. Details from the ensuing investigations are being collected by DMIC. These will be summarized in a technical note for distribution on request at the earliest opportunity. In the interim, preliminary information which was provided to DMIC by NASA/MSC(1) is summarized below to acquaint the reader with the possible hazards associated in subjecting titanium to high stresses in the presence of methanol.

The Ti-6Al-4V tank that failed had been solution treated and aged to meet minimum room-temperature strength requirements of approximately 165 ksi ultimate tensile strength and 155 ksi tensile yield strength. It was fabricated from forged and forged-and-ring-rolled hemispherical end caps and cylindrical sections, respectively, by joining these with circumferential welds. Following welding, the tank received a combined stress-relief annealing and aging treatment of 4 hours at 1050 F in air. Prior to the test in which failure occurred, the tank had successfully passed several pressure tests wherein inhibited water and helium, as well as methanol, was used. The previous test using methanol had involved pressurization to 175 psig. Failure of the tank occurred subsequently after it had been partially filled with methanol and was pressurized, under helium, to 240 psig (equivalent skin stress of 106 ksi). This failure occurred approximately 1 hour and 50 minutes after pressurization. The resulting explosion impacted fragments of the tank against another Ti-6Al-4V tank containing a simulated oxidizer. The impact caused failure of the second tank.

Follow-up studies are now in process at NASA/MSC as well as other laboratories to identify and more carefully define the factors associated with this failure. The initial NASA/MSC investigations included laboratory tests at room temperature using materials from the failed tank as well as control Ti-6Al-4V sheet, i.e., material which had been heat treated to the same strength level but which had not been previously exposed to methanol. Early results from these studies are summarized as follows.

Tests on Material from the Failed Tank

A. Samples exposed to reagent-grade methanol and/or Federal Specification OM-232d methyl alcohol in cyclic loading (6 cycles/minute between stresses of 7 and 140 ksi) failed much more rapidly (e.g., after 86 cycles) than did the same material stressed in air or distilled water (after 1385 and 1269 cycles, respectively) or in a variety of other liquid environments including isopropyl alcohol, Freon MF, and an ethylene glycol-H_2O solution.

B. Sustained loading tests showed that, while this material withstood 75 ksi in methanol for 18 hours, failures occurred in less than 35 minutes at a stress level of 90 ksi. Progressively shorter failure times with increasing stress levels above 90 ksi were indicated.

C. The introduction of a notch ($K_t = 6.0$) accelerated failures on samples exposed in methanol at stresses of 100 ksi and greater.

Tests on Control Sheet

Limited results indicate this sheet is not so susceptible to failure on stressing in reagent-grade methanol as material from the failed tank, e.g., one out of one control sample tested survived a stress of 120 ksi for 48 hours. However, notched samples ($K_t = 6.0$) of control sheet or unnotched samples initially oxidized in air for 4 hours at 1000 F failed after stressing at 120 ksi in methanol in times of less than 65 minutes.

A variety of fluids are being evaluated as possible pressurizing agents for Ti-6Al-4V alloys in this and similar applications. Encouraging results have been obtained with several fluids including Aerozene 50. More definite results will be available in the near future.

In the opinion of DMIC, this effect of methanol may be related to its ability to lower the surface energy of certain metals. This means that, under stress, cracking may take place at reduced stress levels. Methanol is an example of a number of liquids, including water and mercury, that are known as "surface active agents" or simply as "surfactants". The activity of these surfactants varies with their chemical nature and the metal system to which they are exposed. For example, water is a very active surfactant for many materials (including certain steels), but for titanium, water may have little or no effect.

The nature and effects of surfactants on the mechanical properties of metals have been studied extensively in Russia and in the United States by Kramer and Demer(2) and more recently by Rostoker.(3) So far as is known to DMIC, none of this work has been done with titanium or its alloys. However, these experiences suggest that the failures associated with the cracking of Ti-6Al-4V alloy in methanol are not unique to titanium and do occur with other metal systems in an environment of stress and the proper surfactant.

Hot-Salt Cracking

A study has been started at Norair Division of Northrop to determine what effect thermal cycling between 150 F and 450 to 650 F has on the hot-salt (NaCl) stress-corrosion cracking of titanium alloys.(4) Norair is also investigating the effect of fatigue cracks. The study is intended to show whether nucleation, at the elevated temperature, begins again after each low-temperature cycle or to what degree the nucleation time is cumulative.

Work at the National Bureau of Standards shows that the severity of hot-salt cracking of titanium alloys varies with the chloride salt.(5) The severity decreases in the following order: $BaCl_2$, KCl, $CaCl_2$, CsCl, $SrCl_2$, and $MgCl_2$. Cracks were not found in specimens exposed to $MgCl_2$ for 280 days at 800 F. Also, it was shown that the severity of cracking increased with LiCl content in NaCl containing 100 and 1000 ppm lithium.

Cracking in N_2O_4

Bell Aerosystems has published the results of an extensive study of the failure of Ti-6Al-4V alloy tanks containing N_2O_4.(6-9) It was shown that the Ti-6Al-4V is susceptible to stress-corrosion attack when exposed to Specification N_2O_4 (containing no NO). Temperature was shown to be a more dominant factor than stress (pressure). The effect of the pressurant (helium or nitrogen) on cracking susceptibility was negligible. Stress-corrosion cracking occurred at stresses as low as 40 ksi. Variations in fabrication processes did not affect susceptibility. Anodizing, oxidizing, or tumble cleaning with Al_2O_3 (lightly peening) did not improve the stress-corrosion-cracking behavior. Teflon coating was found to be a promising but not completely proven technique for preventing failure. It was found that the 6061 aluminum alloy is an acceptable alternate material. Also, the Ti-6Al-4V alloy did not stress-corrosion crack in the MMH or 50/50 fuel blend at 105 F. The stress corrosion of the Ti-6Al-4V alloy in N_2O_4 was inhibited by the addition of >0.18% water, 7% nitric acid, or >0.06% NO. Silver nitrate did not act as an inhibitor.

As a result of these and other studies, the NO content of N_2O_4 is now specified as 0.4 to 0.8 percent NO. This product is referred to as "Inhibited N_2O_4", and titanium alloys can be used to contain it.(10)

Cracking from Sealants

Preliminary studies conducted at the Air Force Materials Laboratory indicate that fluorine-containing sealants promote stress-corrosion cracking of titanium alloys at temperatures above 350 F.(11)

CORROSION OF ALUMINUM ALLOYS

General

Classes of corrosion inhibitors for aluminum and examples of each class have been discussed in a paper by Kaiser Aluminum.(12) Also listed were inhibitor compositions for controlling the corrosion of aluminum in some 50 aggressive environments.

Five major corrosion problems have been identified by Lockheed-California on naval aircraft operating in the Southeast Asian Theater.(13) These include intergranular corrosion, galvanic corrosion, stress corrosion, filiform corrosion, and erosion of leading edges resulting in pitting corrosion. Corrosion has been minimized in the field by frequent washing with fresh water or with salt water and emulsion solvents where fresh water is not available.

Stress-Corrosion Cracking

The stress-corrosion cracking of aluminum alloys has recently been summarized by DMIC.(14) Included in the report are descriptions of (1) the historical development and growth in awareness of the problem, (2) experimental techniques and test methods, (3) the mechanisms involved, (4) the theory of stress-corrosion cracking, and (5) preventive measures.

The effect of cathodic polarization on the stress-corrosion-cracking behavior of 7075 aluminum alloys in NaCl-acidified $AlCl_3$ has been studied at Alcoa.(15) Over the potential range of -0.75 to 1.3 v (saturated calomel electrode), a maximum in failure time was observed at -0.95 v and a minimum at -1.15 v. Short-time pre-exposure of specimens at -1.15 v significantly shortened the total life of stressed specimens upon subsequent exposure at -0.95 v. These results led to the conclusion that crack initiation was relatively low at -0.95 v, while crack propagation was relatively high at this potential.

Alcoa has developed a test to determine whether aluminum 2219-T851 products have been adequately aged to produce low susceptibility to stress-corrosion cracking.(16) The test involves measuring the potential between a standard calomel half cell and the 2219-T851 test piece after 30 minutes in a $CH_3OH\text{-}CCl_4$ solution containing dissolved 2219 alloy. If the potential difference is -800 mv or more anodic (active), the material is adequately aged.

New research programs on the stress-corrosion cracking of aluminum alloys have been initiated at Rocketdyne and Boeing. The study at Rocketdyne will deal with an investigation of the mechanism of stress-corrosion cracking as it is related to the difference in behavior of the 7075 alloy in the T6 and T73 conditions.(17) The Boeing research is an alloy-development program directed toward producing a high-strength aluminum that is less susceptible to stress-corrosion cracking than the present 7000 series alloys.(18) Additions of silver, zirconium, boron, cerium, and yttrium to the 7075 alloy are being studied.

CORROSION OF FERROUS ALLOYS

Hydrogen Embrittlement

Hydrogen embrittlement of various alloys has been studied at Battelle-Columbus.(19) Cathodic charging experiments indicated that Ti-6Al-4V, Alloy 718, Waspaloy, René 41, and U-212 steel were not susceptible to hydrogen stress cracking, but that the following alloys were susceptible (increasing in the order listed): 17-7PH stainless steel, AISI E-8740 steel, 18Ni maraging steel, AM-355 stainless steel, 17-4PH stainless steel, AISI H-11 tool steel, AISI 4130 steel, and AISI 4340 steel. There was no correlation between the rate at which a material accepted hydrogen and its cracking susceptibility. It was also found that conventional electroplating processes could introduce sufficient hydrogen to cause embrittlement in susceptible alloys and that the plating process introduced more hydrogen into the specimen than did the cleaning process.

Stress-Corrosion Cracking

The stress-corrosion-cracking behavior of two ferritic stainless steels, Types 430 and 434 (430 + 1% Mo), has been investigated at the Climax Molybdenum Company.(20) It was found that these alloys stressed to above the yield point did not stress-corrosion crack in boiling $MgCl_2$ at 284 F, in boiling 55% $Ca(NO_3)_2$ at 242 F, or in boiling 25% NaOH at 232 F. Type 434 alloy was more resistant to pitting in the chloride solution than the 430 alloy. Both alloys heat treated at 1800 F were subject to intergranular corrosion in the $Ca(NO_3)_2$ and the NaOH solutions.

An experimental alloy (45Ni-20Cr-5Mn-0.03C) has been developed at Armco Steel to meet the objectives of (1) good welding characteristics and (2) immunity to stress-corrosion cracking.(21) Manganese was added to improve the welding characteristics and did not affect the stress-corrosion-cracking behavior. Manganese also improved the forging and rolling characteristics.

CORROSION OF NICKEL-BASE ALLOYS

The corrosive effects on superalloys of manganese and lead compounds in JP-4 fuel exhaust gas has been studied at Philips Petroleum.(22) TEL (tetraethyl lead) and CI-2 (methyl cyclopentadienyl manganese tricarbonyl) additions to JP-4 were evaluated with various amounts of sulfur in the fuel and in the presence and absence of sea salt injection in the combustion section. There was considerable variation among the different alloys with fuels of different sulfur content and at different temperatures. Corrosion was more severe in the presence of sea salt, and the additions of manganese or lead compounds had no significant effect on the corrosion of the alloys under these conditions. In general, in the absence of salt, more corrosion was observed with CI-2 additions than without this additive. Attack was more severe with TEL additions under these conditions. Because of the increased corrosion under some conditions, the CI-2 additive was not recommended without extended engine tests.

A study of the high-temperature corrosion of turbine components in gases from sulfur-bearing fuels has been conducted at Westinghouse.(23) The observed corrosion in nickel-base superalloys has been shown to be associated with compounds that can reduce the protective oxide film on metal surfaces. The relative resistance to corrosion appeared to be related to the melting point of the compound formed and to the chromium or aluminum contents of the alloy. Chromium served to prevent the formation of the nickel-nickel sulfide eutectic. These observations suggested that, at the point where the chromium sulfide is formed, the alloy becomes depleted in chromium and is then susceptible to oxidation. Also suggested was that the reaction is autocatalytic in that subsequent oxidation of the sulfide complex releases sulfur for additional formation of sulfides with the base metal. Thus, alloys of higher chromium content are more resistant to attack. However, higher chromium content decreases the hardening action of aluminum and titanium so the selection of gas-turbine alloys becomes a compromise between corrosion resistance and strength level.

Work at TRW has shown that protective coatings of aluminum and chromium-aluminum provide protection of nickel-base turbine superalloys in sulfur-containing fuels.(24) However, if the coating is damaged, the corrosion behavior is dependent upon the base metal only, and normal rapid attack will occur.

A series of small burner tests conducted at General Electric, with 7 different iron-, cobalt-, and nickel-base alloys and 7 different commercial liquid and gaseous fuels, has provided extensive comparisons to aid in the selection of turbine alloys.(25) The long-term tests (6000 hrs) were conducted in the temperature range between 1400 and 2000 F. These laboratory results were then compared with the measured attack in actual turbine parts after 70,000 hrs of field service. The two results compared well, but generally the field corrosion rates were higher than the test burner results.

The reaction of molten NaCl and Na_2SO_4 with oxide films on nickel-base superalloys and nickel at 1650 F has been studied at the Marine Engineering Laboratory.(26) Reactions with Cr_2O_3 and NiO occurred in the presence of carbon (reducing conditions) and not under oxidizing conditions. Half-cell potential measurements were found to correlate well with sulfidation attack. A sustained rise in the half-cell potential was observed whenever sulfidation occurred. The AMS 5391A alloy was attacked by both Na_2SO_4 and Na_2SO_4/NaCl mixtures, while the AMS 5384 alloy was attacked by the mixture only.

CORROSION OF REFRACTORY METALS AND RARE EARTHS

Investigations at Lewis Research Center have shown that both tantalum and columbium are embrittled by hydrogen under conditions simulating a hydrogen-cooled rocket nozzle.(27) A temperature gradient of RT to 3000 F was maintained on the specimens. Quenching rates were 25 to 1500 F per min. Three commercial tantalum alloys and 10 columbium alloys absorbed large quantities of hydrogen and exhibited embrittlement and disintegration similar to that observed with unalloyed tantalum and columbium. On the other hand, hydrogen absorption was less than 1 at.% in Cb-25 at.% Rh and Ta-25 at.% Rh alloys.

The oxidation characteristics of 15 lanthanide metals (atomic numbers 57 to 71) have been examined in detail in dry and moist air at 100 to 800 C in studies conducted at Rensselaer.(28) It was found that oxidation in dry air generally proceeded according to linear or parabolic oxidation kinetics. Cerium, erbium, and terbium oxidized catastrophically at high temperatures. Water vapor generally increased the oxidation rates because of the formation of less protective hydroxide films. This effect was less pronounced at high temperatures where water adsorption was retarded.

The behavior of pure tantalum in a simulated re-entry environment was studied by the Sandia Laboratory.(29) The rate of reaction and mass loss was found to be a function of oxygen content of the gas and the square root of the re-entry model stagnation pressure. The reaction products melted at about 1800 C. The test results showed that Ta_2O_5 is not the only reaction product, but rather that tantalum and Ta_2O_5 are melted together to form a composition near "TaO".

Protective Coatings and Passive Films

A program has been carried out at Aerojet-General to evaluate and develop coatings suitable for the protection of hardware in missile silos that may be contaminated with N_2O_4.(30) Approximately 40 coatings were evaluated. A furfural-base coating was shown to have the best combination of properties. A wholly inorganic system was found to be excellent under dry conditions and where flexibility was not needed. Chlorinated butyl rubber-base coatings also showed promise, but were not yet developed to a point where they can be recommended.

A number of tapes and strippable coatings have been evaluated at Rock Island Arsenal in terms of their corrosion, weathering, and fungus resistance and their adhesion, adhesive transfer, and holding power.(31) The best protection was provided by the plastic tape MIL-T-22085, Type II. A weather-resistant polyvinyl fluoride-backed tape and a strippable vinyl plastisol coating caused corrosion of the metal surfaces.

A study at Edwards Air Force Base has the objective of obtaining detailed information on the composition, structure, and properties of passive films formed on metals exposed to fluorine and certain interhalogen compounds.(32) The metals being studied are nickel, copper, Monel, stainless steels, and aluminum alloys. Preliminary data show apparent fluoride film thickness of up to 25 A in ClF_3 and up to 13 A in fluorine. In particular, the film thickness on aluminum alloys is greater in ClF_3 than in fluorine. In fluorine, increased pressure produced a thicker film on nickel. Anodic polarization experiments in BrF_3 showed that electrochemical reactions are occurring on stainless steel and Monel. The preliminary data indicate that more than one anodic reaction occurs and that chemical passivation alters the anodic reactions.

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