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HOT CORROSIVITY OF COAL CONVERSION PRODUCTS ON
HIGH TEMPERATURE ALLOYS

Final Report

By
Gerald H. Meier

August 29, 1984

Work Performed Under Contract No. AS01-76ET10577

University of Pittsburgh
Pittsburgh, Pennsylvania

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FINAL REPORT

ON

Contract AS01-76-ET-10577

Submitted to

U. S. DEPARTMENT OF ENERGY

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August 29, 1984

1.0 Summary

This report describes research begun under Contract AS01-76-ET-10577, most of which was continued when the same project was continued under a new number, Contract AC01-79-ET-13547. The areas studied are (i) Thermochemistry of High Temperature Corrosion, (ii) Hot Corrosion of Turbine Alloys and Coatings, and (iii) Electrochemistry of Sulfate Melts. A background to the problem of hot corrosion is presented first followed by a description of results obtained in the three areas of this project.

(iii) Deposits on turbine components will likely be by Na_2SO_4 - K_2SO_4 mixtures.

The last point is consistent with the thermodynamics of the Na-S-O and K-S-O systems⁽⁷⁾. At oxygen pressures on the order of one atmosphere the sulfates are stable even at low sulfur pressures.

The difference in melting points of Na_2SO_4 (1157K) and K_2SO_4 (342K) indicates less likelihood of forming liquid deposits of K_2SO_4 at the temperatures of turbine operation (1000-1400K). However, a eutectic exists in the Na_2SO_4 - K_2SO_4 system at 20 mole percent K_2SO_4 and a temperature of 1150K⁽⁸⁾ and potassium pyrosulfate is stable in K_2SO_4 at low pressures of SO_3 at low temperatures (650-750K) resulting in liquid phases⁽⁹⁾. Also, it is observed that CoSO_4 produced by reaction of Co-alloys with SO_3 forms low melting solutions with Na_2SO_4 at temperatures as low as 873⁽¹⁰⁾. Similar low melting phases also occur between the alkali metal sulfates and NiSO_4 . Therefore, it is clear that liquid deposits containing both K_2SO_4 and Na_2SO_4 are of potential interest over the entire operating temperature range of gas turbines.

The presence of Pb, Mo, and V as contaminants may alter the chemistry of deposits and, therefore, influence the corrosion process. All three have been shown to produce detrimental effects in the Na_2SO_4 induced hot corrosion of some alloys. The presence of Mg and Ca in the coal and the use of dolomite and limestone for S removal presents the possibility of MgSO_4 and CaSO_4 being present in deposits on turbine components. The presence of MgSO_4 may be particularly important since it forms low melting mixtures with Na_2SO_4 which are apparently quite corrosive. There is also a high probability of Si being contained in the deposits either as SiO_2 or silicates.

Once a deposit has formed on an alloy surface the extent to which it affects the corrosion resistance of the alloy will depend on whether or not the deposit melts, how adherent it is, and the extent to which it wets the surface. A liquid deposit is generally necessary for severe hot corrosion to occur although some examples exist where dense, thick solid deposits have, apparently, resulted in considerable corrosion. A particularly important example is intense sulfidation/oxidation corrosion of heat exchanger materials in fluidized bed coal combustors associated with CaSO_4 -containing deposits and local conditions of low oxygen pressures⁽¹¹⁾. Work being conducted in the present program has indicated that CaO can be very detrimental to the oxidation resistance of Cr_2O_3 -forming alloys even in the absence of sulfur.

The relationship of adhesion and wetting to hot corrosion has not been studied. Furthermore, the factors which affect the adhesion and wetting behavior of sulfates on metal or oxide surfaces have not been extensively investigated. However, considerable work on the surface tension and wetting behavior of alkali metal carbonates is available in the Russian literature⁽¹²⁻¹⁴⁾. This work has shown that the adhesion and wetting is, of course, affected by the surface tension of the liquid with the contact angle increasing with increasing surface tension. The surface tension was, in turn, influenced by the molecular structure and composition of the liquid and the temperature. It was found that surface tension increased with increasing binding energy and decreasing cation radius for the carbonates of Li, Na, K, and Cs. A similar trend was reported for the sulfates of the same metals⁽¹²⁾. Additives such as oxides were found to decrease the surface tension of the carbonates if their effective cationic dipole moment was lower than that for the cation of the carbonate. The atmosphere was found to have little effect on the

surface tension. The tendency of a given liquid to wet a surface was found to depend on the nature of the substrate; its composition; crystal structure, roughness, etc. The tendency of the liquid salt to wet the surface of an alloy is quite important to its hot corrosion behavior in that it will affect the adhesion of the deposit and, for a given loading of salt, will determine the local deposit thickness. Increased salt thickness has been found to affect the rate of hot corrosion and, depending on the particular mechanism, may accelerate or retard the reaction⁽³⁾. Work in the present program ⁽¹⁵⁾ has indicated that Na_2SO_4 wets different alloys to different extents with one of the important variables being the nature of the surface oxide. For example the extent of wetting was different on two Al_2O_3 -forming alloys, Co-18Cr-6Al and Fe-18Cr-6Al, due to the presence of Co-containing transient oxides in one case and Fe-containing in the other.

Once the alloy surface has been partially or completely wet by the molten salt conditions for severe corrosion may develop. The hot corrosion of virtually all susceptible alloys is observed to occur in two stages: an initiation stage during which the rate of corrosion is slow and a propagation stage in which rapid, often catastrophic, corrosion occurs. During the initiation stage the alloy is being altered to make it susceptible to rapid attack. This alteration may include depletion of the element responsible for forming the protective scale (usually Al or Cr), formation of sulfides in the alloy due to sulfur penetration through the scale, dissolution of oxides into the salt, and development of growth stresses in the scale. This alteration also can result in shifts in salt composition toward more corrosive conditions. The length of the initiation stage varies from seconds to thousands of hours depending on a large number of variables including alloy composition, alloy microstructure, salt composition, atmosphere, temperature, extent of thermal

cycling, salt thickness, specimen geometry, and presence or absence of erosive conditions. In many cases the end of the initiation stage follows the local penetration of the salt through the scale and subsequent spreading along the scale/alloy interface. This situation in which the salt reaches sites of low oxygen activity and is in contact with an alloy depleted in Al or Cr generally leads to the rapid propagation stage. Propagation occurs by one or more of the following mechanisms:⁽³⁾

- (i) Basic fluxing in which the scale is degraded by reaction with oxide ions in the salt,
- (ii) Acidic fluxing in which the scales are degraded by donating oxide ions to the salt, and
- (iii) Sulfidation which involves intense sulfidation of the alloy and subsequent oxidation of the sulfides to produce a thick, non-protective oxide.

2.2 Hot Corrosion of MCrAlY Coatings

A large number of Ni- and Co-base structural alloys form protective scales in high P_{O_2} atmospheres and exhibit correspondingly low oxidation rates. However, the presence of a deposit such as Na_2SO_4 often results in severe or even catastrophic corrosion of the same alloys. The most common solution to this problem is the use of protective coatings such as diffusion aluminide coatings or the MCrAlY overlay coatings (M = Fe, Ni, or Co). The latter coatings are generally considered the most advanced and provide good oxidation resistance and hot corrosion-resistance at temperature of 900° and above. However, severe hot corrosion of turbine vanes and blades has been observed when operating conditions are such that metal temperatures are in the range 600-800°C and the environment results in significant Na_2SO_4 deposition, e.g. marine environments. This form of

corrosion, termed "low-temperature hot corrosion" causes degradation of most alloys but it is particularly detrimental to coatings such as CoCrAlY. The attack is primarily of a pitting type with the oxide in the pits enriched in Cr and Al in alternating layers and depleted in Co. A Co-rich oxide covers pits. There are virtually no sulfides in the alloy and generally no depletion of β -CoAl below the oxide pit. A thin zone enriched in Al and S and containing O is found at the oxide/alloy interface. Small amounts of water-soluble Co have been observed on corroded coatings.

Several laboratory and burner-rig studies have been carried out to ascertain the effect of environmental and alloy variables on the extent of this form of corrosion and to determine the degradation mechanism. These studies have shown that this form of corrosion can be reproduced by having SO_3 partial pressures in the gas to form a liquid Na_2SO_4 - CoSO_4 solution. Barkalow and Pettit⁽¹⁶⁾ were able to reproduce the corrosion morphology at 700°C by exposing CoCrAlY coatings with Na_2SO_4 deposits to oxygen containing SO_3 at pressures greater than 10^{-4} atm. The rate of degradation was found to be proportional to the SO_3 pressure and negligible in the absence of SO_3 .

Shores and Luthra⁽¹⁷⁻¹⁸⁾ studied the effect of SO_3 on the corrosion of Na_2SO_4 -coated Co-Cr, Co-Al, Co-Cr-Al, and Ni-Cr alloys at temperatures between 600 and 900°C . All of the alloys were heavily attacked at temperatures between 600 and 750°C under conditions where a Na_2SO_4 - CoSO_4 liquid formed. The CoCrAl alloys exhibited the pitting morphology described above while the Co-Cr alloys formed similar morphologies with Cr concentrated in the pit and a layer containing Cr and S at the pit/alloy interface. The Co-Al alloys exhibited a more uniform corrosion front with Co oxides at the scale/gas interface and a zone rich in Al and S at the corrosion front. The Ni-Cr alloys⁽¹⁸⁾ corroded in a uniform manner but the

corrosion morphology was not described in detail.

Aprigliano⁽¹⁹⁾ exposed a range of Ni- and Co-based coatings at 704°C in a burner rig using fuel containing 1% S and air containing 20 ppm sea salt. NiCrAlY underwent less corrosion than CoCrAlY and for the CoCrAlY coatings increasing the Cr-content from 20 to 30% at 12Al-0.5Y resulted in improved resistance. Increasing the Y content to 2% resulted in poorer resistance.

While there is general agreement that SO_3 partial pressures high enough to produce liquid Na_2SO_4 - CoSO_4 (or NiSO_4) solutions are required to initiate severe hot corrosion at temperatures between 600 and 800°C some uncertainty still exists regarding the detailed corrosion mechanism. Barkalow and Pettit⁽¹⁶⁾ propose that the liquid sulfate penetrates the Al_2O_3 scale at cracks. The P_{O_2} at the salt alloy interface is lowered by oxide formation and since the transport of O_2 through Na_2SO_4 is slow relative to that for SO_3 the equilibrium

results in locally high pressures of SO_2 . This environment can then result in the selective removal of Al from the alloy by the formation of a Na_2SO_4 -soluble species, perhaps Al sulfite. The Al is then reprecipitated as Al_2O_3 in regions of the salt nearer the liquid/gas interface where the P_{O_2} is higher. The Al-depleted alloy is converted to non-continuous Cr_2O_3 in situ, and the Co dissolves into the liquid and diffuses to the liquid/gas interface forming Co-oxide or sulfate.

Smeggil⁽²⁰⁾ suggests that the corrosion morphology may be reproduced in the absence of SO_3 in the gas phase if the coatings are exposed to brief thermal

excursions and transient reducing conditions and proposes this as an alternate mechanism. Laboratory hot corrosion experiments in which specimens were periodically cycled to 1300°C for 30 seconds in carbonaceous material produced corrosion morphologies similar to those observed in marine turbines. Smeggil does not offer a mechanism for the degradation produced by the high-temperature excursions.

Luthra and Shores⁽¹⁸⁾ suggest that transient Co- or Ni-oxides on Co-Cr or Ni-Cr alloys react with SO_3 to form CoSO_4 or NiSO_4 which form liquid phases with Na_2SO_4 and that the high corrosion rates are due to the rapid dissolution of Co in Ni. It is proposed that the rapid removal of the base metal continually undercuts Cr_2O_3 particles and prevents them from forming a protective layer.

Further work in identifying the mechanisms of low-temperature hot corrosion is needed, particularly since the deposits and atmospheres likely in systems fueled by coal conversion products almost certainly lead to similar forms of corrosion.

3.0 Review of Results of This Program

The research conducted under Contract ASOI-76-ET-010577 and continued under Contract DE-ACOI-79-ET-13547 concentrated on three areas.

1. Evaluation of Thermochemistry of High Temperature Corrosion.
2. Hot Corrosion Studies of Turbine Alloys and Coatings.
3. Electrochemistry Studies of Sulfate Mixtures.

The results of these efforts will be briefly described.

3.1 Evaluation of Thermochemistry of High Temperature Corrosion

The corrosion problems associated with all coal conversion processes are

complex involving the reaction of multicomponent alloys with gases containing two or more reactive components. The situation is often complicated by the presence of liquid or solid deposits which form either by condensation from the vapor or impaction of particulate matter. An important tool in the analysis of such problems is, of course, equilibrium thermodynamics which, although not predictive, allows one to ascertain which reaction products are possible, whether or not significant evaporation or condensation of a given species is possible, the conditions under which an oxide scale can react with a condensed deposit, etc. The complexity of the corrosion phenomena usually dictates that the thermodynamic analyses be presented in graphical form.

Work on this project resulted in the publication of an extensive set of condensed phase stability diagrams for most of the metal-sulfur-oxygen systems of interest in high temperature corrosion and vapor species diagrams for the corresponding metal-oxygen and metal-sulfur systems⁽⁷⁾. A similar set of diagrams for metal-carbon-oxygen and metal-nitrogen-oxygen systems was also published⁽²¹⁾.

3.2 Hot Corrosion Studies of Turbine Alloys and Coatings

3.2.1 Hot Corrosion of Coating Alloys

Earlier work in this and other programs had shown that uncoated-structural alloys would not have sufficient hot corrosion resistance for most components in systems burning coal conversion products. Therefore, the hot corrosion of coating alloys was studied with particular emphasis on the problem of low-temperature hot corrosion. The results of this study and a parallel NASA-sponsored study (Grant No. NSG-3214) have recently been published⁽²²⁾. The major findings of this study were as follows. The reaction of Co-Cr, Ni-Cr, Co-Cr-Al, and Ni-Cr-Al alloys with

Na_2SO_4 in the presence of SO_3 at temperatures between 700 and 750°C results in the formation of liquid $\text{CoSO}_4\text{-Na}_2\text{SO}_4$ or $\text{NiSO}_4\text{-Na}_2\text{SO}_4$ deposits on the alloy surface. The formation of Cr_2O_3 and/or Al_2O_3 below this deposit results in a locally low P_{O_2} and increased P_{S_2} and P_{SO_2} . These conditions can prevent protective oxide formation either by sulfide formation in the alloy which localizes the Cr and/or Al in discrete particles or by acid fluxing involving a reaction between Al_2O_3 or Cr_2O_3 and SO_2 to form a salt-soluble species and subsequent reprecipitation as porous oxides in regions of higher P_{O_2} . These processes may occur simultaneously but a given alloy generally exhibits features of predominantly one type. In the present study the Ni-Cr and Ni-Cr-Al alloys and, to some extent, the Co-Cr alloys showed features indicative of the sulfidation mechanism whereas the morphology for the Co-Cr-Al alloy was more consistent with the acid fluxing mechanism predominating.

Either mechanism frees Co or Ni which dissolves in the sulfate melt and diffuses outward forming an oxide layer at the salt/gas interface. It is not believed that this dissolution process is responsible for the rapid attack of alloys in this temperature range because (i) the hot corrosion of pure Co was not accelerated by SO_3 pressures which rapidly degraded Co-Cr-Al and (ii) the lower-Co, high-Al γ -phase was preferentially attacked in Co-Cr-Al.

Several other conclusions may also be drawn from the experimental results

- (a) The presence of Al appears to result in more severe degradation as evidenced by higher SO_3 pressures being required to degrade Co-27Cr as compared with Co-18Cr-6Al and, at a given P_{SO_3} , a Co-18Cr-6Al alloy being more severely attacked than a Co-20Cr alloy.

(b) Nickel base alloys exhibit similar corrosion morphologies to these formed on Co-base alloys. However, higher SO_3 pressures are required to produce comparable degradation on Ni-Cr-Al as compared with Co-Cr-Al. This may be due to more rapid liquid formation on the Co-base alloy or the absence of the Al-rich β -phase in the nickel base alloys.

(c) A pitting type morphology may be produced by a number of different corrosion mechanisms.

3.2.2: Hot Corrosion Due to Solid Deposits

The severe corrosion sometimes observed in fluidized bed combustors which has been suggested to be the result of CaSO_4 deposits, which should be solid, has raised the question of the extent to which solid deposits can cause hot corrosion. Therefore, the effects of CaSO_4 and CaO on the high temperature corrosion of several Cr_2O_3 -and Al_2O_3 -forming alloys were studied. Exposures of preoxidized alloys were carried out both in crucible tests and with thin coatings of CaSO_4 or CaO in pure O_2 . The CaSO_4 deposits were rather innocuous to all of the alloys tested. However, CaO resulted in severe corrosion of Cr_2O_3 -forming alloys such as Ni-30Cr. Two mechanisms have been identified. At temperatures around 900°C the CaO reacts quickly with $\text{CrO}_3(\text{v})$ which evaporates from the Cr_2O_3 scale and forms CaCr_2O_4 . This "gettering" effect results in continued evaporation and in, some cases, a scale consisting essentially of NiO replaces the Cr_2O_3 . This situation would clearly be dangerous in gases containing a significant sulfur pressure. The second mechanism occurs at temperatures above the eutectic temperature in the $\text{CaO-Cr}_2\text{O}_3$ system. In this case reaction between the two oxides produces liquid solutions and catastrophic corrosion (a 2mm piece of pure Cr was completely consumed in several hours at 1050°C). Alloys forming Al_2O_3 scales

should be less susceptible to this form of attack due to the absence of volatile oxides and low melting phases in the $\text{CaO-Al}_2\text{O}_3$ system. However alloys such as Ni-Cr-Al were attacked severely at 1050°C , due to liquid formation by reaction between CaO and Transient Cr_2O_3 .

3.3 Electrochemistry Studies of Sulfate Mixtures

Previous work in this study determined the effect of various contaminants (K_2SO_4 , SiO_2 , TiO_2 , MoO_3 , MgO etc.) in changing the activity of Na_2O (equivalent to oxide ions) in Na_2SO_4 . The extent of these effects and their time dependence was studied. In the present period the solubility of SiO_2 in Na_2SO_4 was determined as a function of Na_2O activity. These measurements are important since SiO_2 will often be present in sulfate deposits on hardware, coming either from the alloys or contaminants in the atmosphere. Furthermore, protective SiO_2 scales on coatings or Si-based ceramics may be attractive for some high temperature applications in coal conversion systems.

The solubilities were measured by the technique used by Rapp and co-workers^(23,24) Na_2SO_4 at 1200K as a function of Na_2O activity and P_{O_2} . The Na_2SO_4 was contained in a silica crucible and silica powder added. The Na_2O activity was controlled by the SO_3 pressure in equilibrium with the bath and was measured by the cell

while the P_{O_2} in the melt was monitored by the cell

Samples removed from the saturated Na_2SO_4 were analyzed for Si by atomic absorption analysis. The results of these measurements are plotted in Figure 1 along with the earlier data of Stroud and Rapp for Al_2O_3 and Cr_2O_3 solubilities. It is seen that SiO_2 has extremely low solubility in acid melts (low $a_{\text{Na}_2\text{O}}$). This suggests that SiO_2 -forming alloys should be quite resistant to hot corrosion by acid sulfate deposits.

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Figure 1 Solubilities for Al_2O_3 and Cr_2O_3 in Na_2SO_4 as Function of $a_{\text{Na}_2\text{O}}$ for $p_{\text{O}_2} = 1$ atm from work of Stroud and Rapp and for SiO_2 from the present study (solid points).