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EROSION/CORROSION NEWSLETTER
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A.V. Levy

August 1982

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EROSION/CORROSION NEWSLETTER

(Excerpted from LBL-13840)

A. V. Levy

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Berkeley, California 94720

August 1982

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Contract No. DE-AC03-76SF00098.

b. Erosion-Corrosion Wear Program*

Alan V. Levy, Investigator

Introduction. The degradation of materials subjected to streams of gases containing small solid particles, $< 600 \mu\text{m}$, moving at velocities $< 90 \text{ m/s}$ is being studied. The conversion of coal to clean, combustible gas and the direct combustion of pulverized coal in fluidized bed combustors subjects the materials containing these reactions to erosive forces. The mechanism of the erosion must be understood in order to prevent unacceptable degradation of the containment wall surfaces.

In 1981 several aspects of the erosive behavior of metals were investigated. The effect of the eroding particle characteristics on the erosion mechanism in ductile alloys was determined. The erosive behavior of hard metals was studied as was the effect of various strengthening heat treatments in low alloy steels. Erosion rates at elevated temperatures were determined for aluminum and steel alloys. An initial model of erosion embodying a mechanism of forging-extrusion of metal platelets was developed.

The corrosion of stainless steels whose surfaces were modified by doping with aqueous nitrate salts of active elements applied to the surface was determined. Modification of the reactivity of turbulent gases in curved pipes by injection of water vapor in the unstable boundary layer was studied.

1. THE EFFECT OF HEAT TREATMENT OF STEEL ON EROSION BEHAVIOR†

Tom Foley and Alan Levy

The use of heat treatments to tailor the engineering performance of alloys for various service environments is common. The effects of such heat treatments on the erosion of structural materials must be determined in order to design long life components for coal conversion and combustion systems. In earlier work, it was determined that plastic deformation of metals had a direct relationship to erosion resistance. The effects of various heat treatments on the nature of the flow of alloys and, hence, their erosion resistance was determined.

Plain carbon 1020 steel was tested in its pearlitic and spheroidized microstructures above and below its ductile brittle transition temperature; low alloy 4340 steel was tested in the as-quenched, 200°C and 500°C temper conditions and spheroidized annealed and 304 stainless steel was

tested in the as-rolled and fully annealed conditions. The alloys were eroded by a stream of Al_2O_3 particles, $140 \mu\text{m}$ diam. average size at 30, 60, 90 m/s velocity at impingement angles of 30° and 90° in air at 25°C .

The erosion rates decreased with increasing elongation and increased with increasing hardness, further verifying earlier work in this project. The observed mechanism of erosion in all of the steels tested was the formation and removal of small platelets of metal by a forging-extrusion mechanism. The four 4340 steel heat treatments all showed approximately the same erosion rate. They had similar tensile elongations, but major differences in tensile strength and hardness indicating that these properties had little effect on erosion behavior. All of the alloys reached steady state erosion in 60 to 200 grams of impacting particles, indicating that erosion rates of steels can be determined in relatively short time tests.

The 304SS with the higher strain hardening coefficient (0.45) reached steady state erosion after 30 grams of particles while the 1020 steel with the lower strain hardening coefficient (0.15) reached steady state erosion after 100 grams of particles. This behavior supports the mechanism of erosion developed earlier - that a cold-worked region develops beneath the eroding surface that enhances the erosiveness of the impacting particles, behaving as an anvil in the forging-extrusion of platelets. Figure 1 shows this effect.

* * *

†Brief version of LBL-13745.

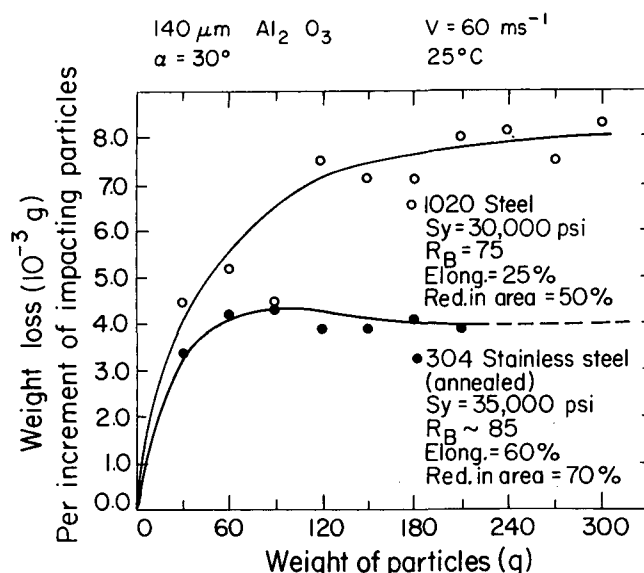


Fig. 1. Effect of strain hardening coefficient of 1020 steel and 304SS on onset of steady state erosion. (XBL 8110-1395)

*This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

2. EROSION OF HARD METALS†

Tom Bakker and Alan Levy

The use of protective coatings on structural metals to enhance their wear and/or corrosion resistance has increased considerably in recent years. Many new coating systems and insert materials for critical areas of coal conversion valves, pumps and other components are now becoming available whose resistance to erosion by solid particles must be determined.

In this project several materials were tested that used the hard metals SiC, WC and Si₃N₄ with various binders and the oxide Cr₂O₃. The materials were fabricated into thin coatings by chemical vapor deposition (CVD), detonation gun spraying and plasma spraying on steel and graphite substrates or by hot pressing into solid bodies suitable for valve inserts. The materials were eroded by SiC particles, 200 μm diam, in an air stream at 30 m/s at 25°C. Impingement angles of 30°, 60° and 90° were used. Erosion rates and mechanisms of erosion were determined and related to the composition and microstructure of the hard metals.

The materials tested were basically hard metal particles bonded with either cobalt or silicon. They all eroded in a brittle manner, i.e., the erosion rate was greater at an impingement angle of 90° than at 30°. The rates of erosion of the

materials varied over a wide range (Fig. 1). Comparative steady state erosion rates for steel alloys range from 0.5 to 2.0 g/g. The most erosion resistant material was CVD deposited CNTD silicon carbide bonded with silicon that had an extremely fine grain size, ~1000 Å diam. The grain size was so fine that the angle of impingement effect observed on all of the other materials tested and, generally, on all brittle materials was not in evidence.

The mechanism of erosion for all of the materials tested was microscopically observed to be cracking and chipping of micron size pieces of material. Hardness did not directly relate to erosion resistance, although the hardest material was CNTD SiC which had a DPH of 3266. The results support the conclusion from earlier work that a fine grain size and low level of porosity enhance erosion resistance of brittle materials. The rates of erosion of the materials reached steady state values after only a few grams of erodent at most had impacted them, also conforming to earlier studies of the erosion of brittle materials. The erosion rates of the hard metals in most cases exceeded those of low alloy and austenitic stainless steels.

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†Brief version of LBL-13801.

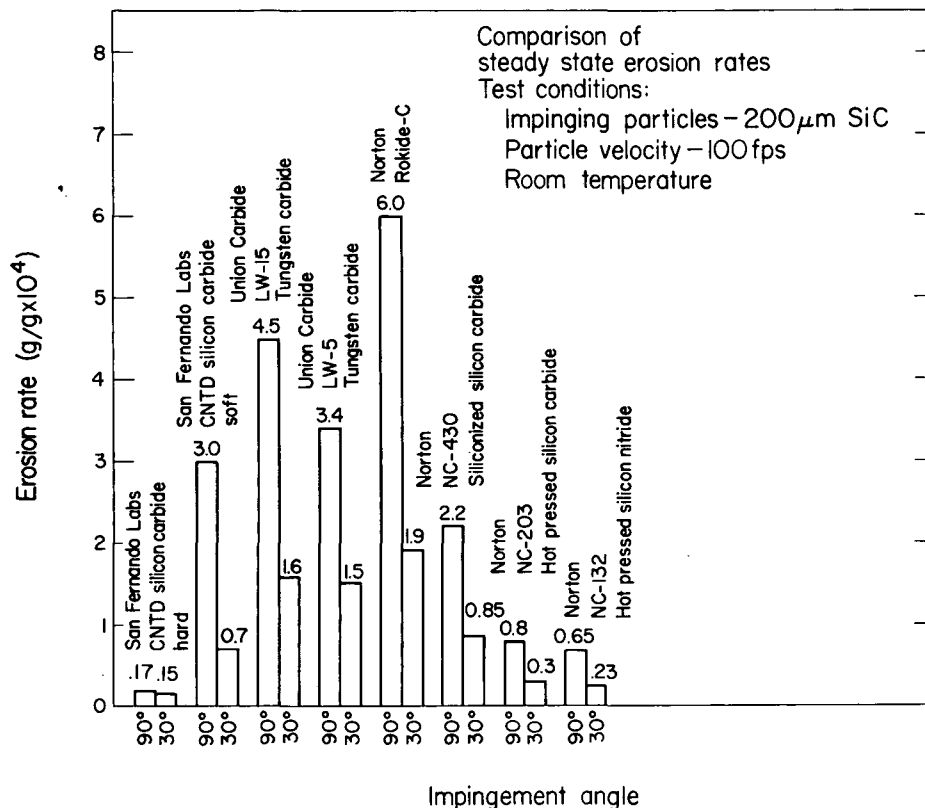


Fig. 1 Comparison of steady state erosion rates of hard metals.

(XBL 818-11438)

3. EFFECT OF PARTICLE VARIABLES ON EROSION OF DUCTILE METALS[†]

Pauline Chik and Alan Levy

The erosion of materials is dependent upon their erosion resistance and the erosion conditions. The effect of variations in the impacting particles on the erosion of ductile metals was investigated to determine how sensitive erosion was to this major variable. In actual service involving solid particle impact erosion, the particles are generally of mixed composition, size, shape and properties. Erosion testing in the laboratory usually uses single composition particles of a comparatively narrow range of sizes and shapes. Whether this discrepancy should be of concern was determined in this project.

Five different minerals were obtained of an average particle size of 200 μm diam. They ranged in hardness from a VHN of 115 for calcite and 300 for apatite to 700 for SiO_2 , 1900 for Al_2O_3 and 3000 for SiC . Steel shot of 200 μm diam, in both angular and spherical shapes was also used. All tests were made at 75 m/s and impingement angles of 30° and 90° at 25°C. Steady state erosion rates were determined and the mechanism of erosion was studied microscopically for a 1020 plain carbon steel target material.

Angular shaped particles above a minimum hardness of approximately VHN = 700 had the same erosivity on 1020 steel (Fig. 1). This is an important finding in that the test particles need not specifically duplicate particles in real environments, but only need be in their general hardness range if below VHN = 700 and can be at any hardness level if above VHN = 700. Thus, the highly reproducible SiC or Al_2O_3 particles will give usable erosion rates for actual service particles of hardness ranging from that of sand upward.

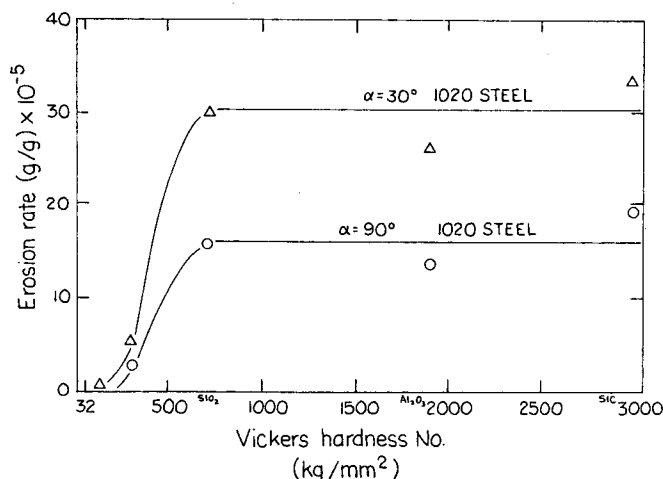


Fig. 1. Effect of eroding particle hardness/integrity on erosion behavior of 1020 steel.
(XBL 8110-7180)

It was also determined that it was not the hardness, per se, that determined the erosivity of the particles, but rather their physical integrity. The softer minerals, calcite and apatite, fractured on impact and could not carry sufficient kinetic energy to the target surface to cause erosion at the higher rates of the stronger minerals. The angular steel shot eroded the 1020 steel at almost four times the rate of the spherical steel shot of nominally the same particle size.

* * *

[†]Brief version of LBL-13802.

4. ANALYTICAL MODEL OF EROSION BEHAVIOR OF DUCTILE METALS[†]

Jolen Flores and Alan Levy

Mounting evidence to support the concept of the erosion of ductile metals by a forging-extrusion mechanism has prompted the development of an analytical model based on that concept to predict erosion of structural alloys. The properties of a material that affect its plastic deformation and surface heating as the result of adiabatic shear deformation and friction must be incorporated into expressions that describe the generation of small platelets of target material. A statistical treatment is also required to account for the average number of impacts necessary to have platelets reach their fracture strain.

Forging equations were utilized and balanced with the energy available to the target material for plastic deformation and that which is used to generate local heating of the near-surface region. Additional kinetic energy for impacting particles is assigned to the subsurface cold working of the target material. The surface temperature during erosion is arrived at by balancing the heat generated by plastic deformation and friction with the heat loss due to convection between subsequent particle impacts. An impact angle of 90° was used.

An erosion model based on forging equations was developed and coupled with basic heat transfer equations. These were in turn coupled with a simple statistical analysis. The predicted erosion rates exhibit the same general profile when plotted against the number of eroding particles as observed experimentally. The temperatures developed on the surface fall within the range of the recrystallization temperature, as postulated from microscopic observation of the eroded surface.

* * *

[†]Brief version of LBL-13803.

5. PREDICTION OF PARTICLE LADEN LIQUID TWO PHASE FLOW[†]

Farzad Pourmahdi and Joseph Humphrey

In order to fully understand the erosion of metals exposed to liquid-solid particle slurry streams, it is necessary to determine the fluid

mechanics of the two-phase flows. In addition to conducting experiments to determine the velocities and positions of the particles in turbulent flow through curved ducts it is important to develop a numerical calculation scheme for predicting such flows. The purpose of this investigation was to develop the model and, subsequently, to use it to predict two-phase flow in laser doppler velocimeter (LDV) monitored flow experiments.

A numerical calculation code, SPLATS (Solid Particles, Liquid and Turbulence Solver) has been developed for predicting turbulent two phase, solid particle-liquid flows in straight and curved two-dimensional channels. Predictions of the solid particle-liquid slurry flow characteristics of various documented cases in the literature show good agreement with the reported experimental results.

* * *

†Brief version of LBL-13808.

6. BOUNDARY LAYER CONTROL BY MEANS OF STRONG INJECTION†

Ruey-Jen Yang and Maurice Holt

The gas mixture produced by a coal gasifier contains components that have serious corrosive effects on the boundaries of the pipe flow system. Injection of a protective gas into the turbulent boundary layer of the coal gas products in a direction parallel to the wall markedly reduces the corrosivity of the inviscid gas stream. The interaction between the injected gas and the product gas pipe flow is investigated analytically; it is an example of the so-called wall jet problem.

The model adopted is that of a two-dimensional incompressible, turbulent free-mixing layer. The corrosive gas, H_2S , forms the upper fast moving stream. The injected protective H_2O vapor in the lower stream is bounded by the solid wall of the pipe. The flow geometry and the development of the mean velocity, U_1 , and concentration profiles of the H_2S , M_i , are shown in Fig. 1. The wall jet flow can be divided into three distinct regions. In the first region, farthest upstream, the upper main stream interacts with the lower injected stream in a free mixing process with a boundary δ_f away from extension of the splitter plate. A turbulent boundary layer develops along the pipe wall bounding the lower stream at a distance δ_w from the wall. In the second region the lower half of the free mixing layer interacts with the wall turbulent boundary layer. In the third region stream mixing has been completed and all diffusion takes place in a thickened turbulent boundary layer. The H_2S initially reaches the pipe wall at this point.

The calculated ratio of the distance from the H_2O injection point to where the mass concentration of H_2S diffuses to the wall surface to the slot height of injection is of the order of 100 slot heights for a given velocity ratio of two

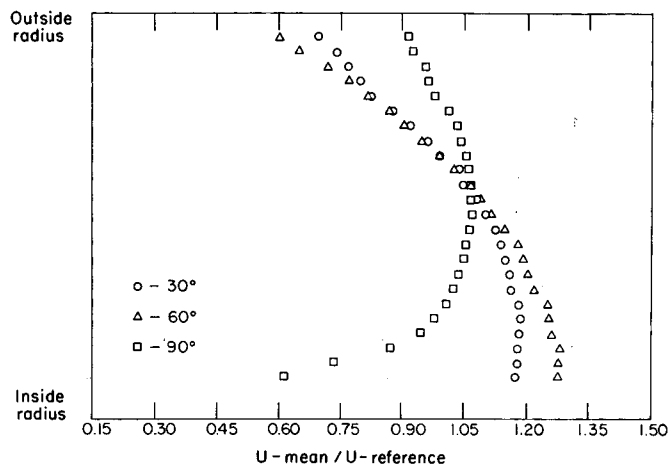


Fig. 1. Streamwise velocity profiles at various stations in a 90° bend. (XBL 8201-4404)

free streams in the mixing layer. Additional distance down the tube wall is required before the partial pressures of oxygen and sulfur reach sulfidizing conditions. This additional distance is estimated to be another ~ 100 slot heights. Thus, injection of water vapor through a 1-cm slot will provide protection against oxidation of the pipe for up to two meters from the point of injection. This distance could make boundary layer gas injection a practical method for protecting surfaces against sulfidation in coal conversion systems.

* * *

†Brief version of LBL-13807.

7. OXIDATION BEHAVIOR OF SURFACE DOPED 304SS WITH MODIFIED COMPOSITION†

Ed Yaniv, Richard Gray, Don Boone, Alan Levy

The purpose of this study was to determine what effect the minor alloying elements manganese and silicon in surface doped stainless steels have on corrosion resistance. This knowledge should be helpful in understanding why the surface application of yttrium to 304SS so improves its high temperature oxidation resistance.

Specimens of 18Cr-8Ni steels were prepared with and without Mn or Si additions and were doped by applying yttrium nitrate in a water solution to polished specimens that had been heated to 500°C in air for 5 minutes. The doped specimens were next heated for 30 minutes at 500°C in air, cooled, and exposed to cyclic oxidation tests for 20 hr/cycle at 1000°C.

Only the 18Cr-8Ni steels that contained silicon had their elevated temperature oxidation resistance enhanced by surface doping with yttrium. The commercial 304SS and the alloy containing only silicon were improved while the 18Cr-8Ni steel with neither Mn nor Si, and the one with only Mn, had their

scales spall on the first thermal cycle, as did the undoped 304SS.

* * *

†Brief version of LBL-13810.

Work in Progress

1. ELEVATED TEMPERATURE EROSION OF ALUMINUM AND STEEL ALLOYS

Jennifer Paterson and Alan Levy

The erosion of materials by particles in coal conversion and utilization systems occurs at elevated temperatures. Testing of these materials at service temperatures in order to determine rates and mechanisms of erosion requires close control of temperature, flow rates, and corrosion conditions. Tests on a newly constructed elevated temperature test device of the required precision have been initiated.

Specimens were exposed to 240 μm diam SiC particles at a velocity of 30 m/s at temperatures up to 800°C. The erosion weight loss of both the 1100-0 aluminum and 310SS were similar functions of the test temperature. Both alloys are FCC single phase metals but they have great differences in strength, modulus of elasticity and other properties. As the temperature was increased to a homologous temperature of approximately 0.15, the erosion rate decreased. At a homologous temperature of 0.3, the highest erosion rate was measured for both alloys, being considerably greater than the room-temperature rate and probably due to changes in the plastic deformation characteristics of the alloys and the actual surface deformation temperature. At a still higher homologous temperature of 0.5 for both alloys, the erosion rates had decreased to the lowest levels in the test series.

At the highest test temperature, considerable amounts of SiC were detected embedded in the surface of the specimens. This SiC could account for the low weight loss measured. Microscopic cross sections and surface observations both showed that the erosion of ductile metals had occurred by the characteristic mechanism of platelet formation.

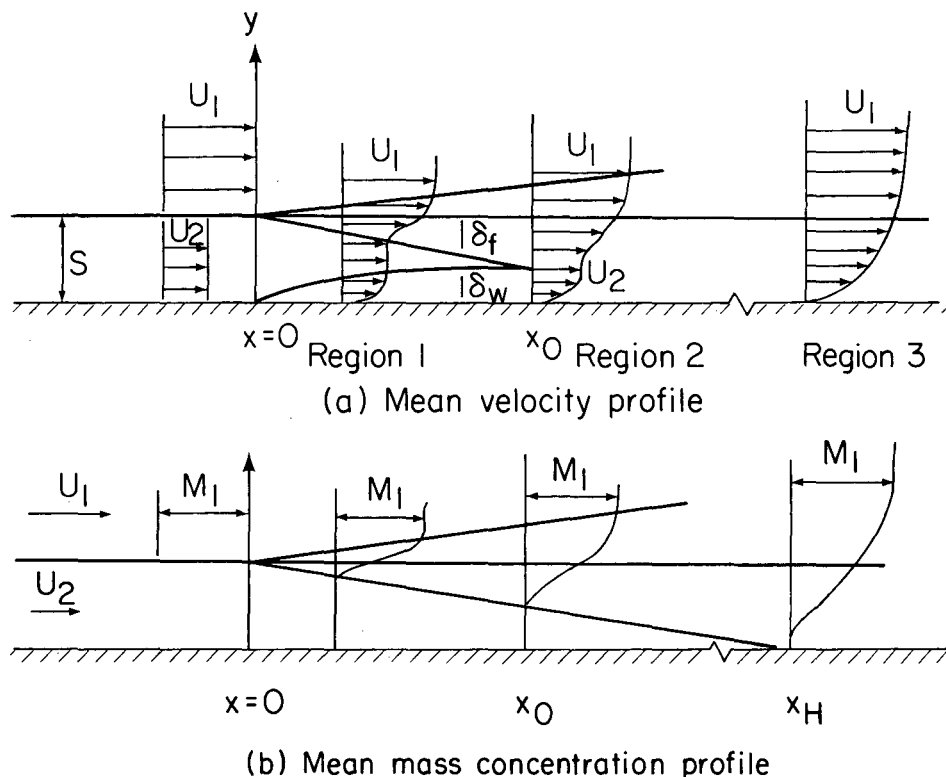


Fig. 1. Behavior of a protective gas boundary layer in a reactive gas flow passage (see next page). (XBL 8201-4403)

2. LASER DOPPLER VELOCIMETRY MEASUREMENTS OF GAS/PARTICLE FLOWS IN A 90° CURVED DUCT

Peter Turi and Jolen Flores

The apparatus designed for the investigation of particle laden air flow in conduits using laser doppler velocimetry (LDV) is now operational and is being used to acquire data relevant to erosion/corrosion during coal gasification. The purpose of this work is to test recently developed analytical models that predict flow of this type in curved ducts.

Velocity measurements were made in the stream-wise and radial directions using atomized mineral oil for particles. Figure 1 shows the profiles at various centerline stations in the 90° bend. The bend causes a gradual transition from a uniform flow (up-stream) to one resembling a potential vortex. This tendency, evident in the 30° and 60° profile, was predicted in earlier, analytical modeling work in this program. The 90° profile shows the influence of the secondary flow. Measurements in the radial direction have quantified this phenomenon. These data agree with the predictions made earlier. A radially inward flow on the walls and a radially outward flow at the centerline have been observed.

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2. A Levy and P. Chik, "Effect of Particle Variations on the Erosion of Ductile Metals," LBL-13802.
3. J. Flores and A. Levy, "Analytical Model of Erosion of Ductile Metals," LBL-13803.
4. T. Foley and A. Levy, "The Effect of Heat Treatment on the Erosion Behavior of Steels," LBL-13745.
5. R. Yang (Ph.D. thesis), "Boundary Layer Control by Means of Strong Injection," LBL-13807.
6. F. Pouramahdi (Ph.D. thesis), "Turbulence Modelling of Single and Two Phase Curved Channel Flows," LBL-13808.
7. E Yaniv, R. Gray, D. Boone, and A. Levy, "Oxidation Behavior of Surface Doped 304SS with Modified Composition," LBL-13810.

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1. A. Levy, "Erosion Behavior in Gasification and Liquefaction Piping and Vessel Environments," ASM Regional Conference, Oak Ridge, TN, May 12, 1981.
2. M. Landkof, D. Boone, D. Whittle, A. Levy, "The Effect of Surface Doping on the Oxidation of Chromia Former Alloys," Gordon Research Conference, New London, NH, July 20, 1981.
3. A Levy and P. Chik, "Effect of Particle Variations on the Erosion of Ductile Metals," AIME-TMS Meeting, Louisville, KY, October 15, 1981.
4. T. Foley, "The Effect of Heat Treatment on the Erosion of Alloy Steels," AIME-TMS Meeting, Louisville, KY, October 15, 1981.
5. A. Levy, "Gas and Liquid-Solid Particle Erosion," Exxon Corporate Research Lab seminar, Linden, NJ, October 9, 1981.
6. A. Levy, "Erosion Behavior of Hard Metals," Union Carbide Corp. Coatings Service Department Research Lab seminar, Indianapolis, IN, October 15, 1981.
7. A. Levy, "Erosion Behavior of Materials," DOE-EMAC seminar, Germantown, MD, October 16, 1981.
8. A. Levy, "Erosion Behavior of Materials," Combustion Engineering Corp. Metallurgy Research Lab seminar, Chattanooga, TN, October 19, 1981.

c. Coal Liquefaction Alloy Test Program*

Alan V. Levy, Investigator

Introduction. The erosion behavior of steel alloys used to construct the slurry flow containment components of coal liquefaction systems is being investigated in this program. The effects on the erosion of steels of flow variables such as particle composition, size, and solids loading; temperature; flow passage geometry; the viscosity and lubricity of the carrier liquid and the velocity and angle of impingement of the particles were determined. The steel alloys selected for testing are those that are commonly used in chemical process plant piping systems. They include: A53 and A106 plain carbon piping steels; 2 1/4 Cr-1Mo, 5Cr 1/2 Mo, 9Cr-1Mo chromium bearing steels and types 410, 304, 316, and 321 stainless steels. The particles that have been used in the slurries have been pulverized coal and SiC. The liquids tested include water, kerosene, creosote oil, hexadecane, and SRC process solvent.

1. EROSION BEHAVIOR OF ALLOYS IN COAL SLURRIES†

Greg Hickey and Alan Levy

The transport of coal using nonaqueous liquid carrier fluids in coal conversion and combustion systems causes erosion to occur on the containment walls of piping, pumps, valves and instrumentation probes. The purpose of this project was to determine the effect of flow variables on the erosion behavior of several steel alloys used in chemical process plant components. The relative performance of the steels tested was also determined.

A slurry pot tester was used in the tests performed. It rotates 3 mm diam by 5 cm long specimens held at the ends of arms on a central rotating shaft in a 3 liter tank of test slurry. Baffles on the tank's inside wall keep vortex flow patterns from occurring as the result of the stirring action. The tests were performed for 2 and 5 hours with periodic weight loss measurements of the test rods. The tester was filled with slurries of various liquids and varying coal content and size and operated at temperatures from 25°C to 175°C. Metallographic analysis of the eroded specimens was conducted after the test exposures to observe the mechanism of erosion.

The effect of particle size and solids loading on the erosion of A-53 mild steel and 304SS showed that more particles and larger size particles resulted in higher erosion rates, particularly at higher velocities. The velocity exponent was also

greater for the larger particle size coal, 3 for -30 mesh coal compared to 2 for -200 mesh coal.

The effect of alloy composition, morphology, hardness, strength and ductility could not be related to the relative erosion rate of the nine steel alloys tested. The A53 mild steel and 304SS consistently eroded at higher rates than the other steels tested. Generally, increasing chromium content of the alloys resulted in lower erosion rates. The smaller grain size of the A106 mild steel caused a marked reduction in the erosion rate compared to the larger grain, same composition A53 mild steel. A variation of hardness of the steels from R_B 72 to R_B 97 could not be related to their erosion rates.

The effect of the temperature of the slurry on the erosion of several alloy steels is shown in Fig. 1. The higher erosion rates of the A53 mild steel and the 304SS compared to those of the other alloys tested was consistent for all variables tested, not just the temperature. The decrease in the erosion rate at the 94°C temperature compared to the 25°C tests is the result of the change in the nature of the slurry and the ductility of the

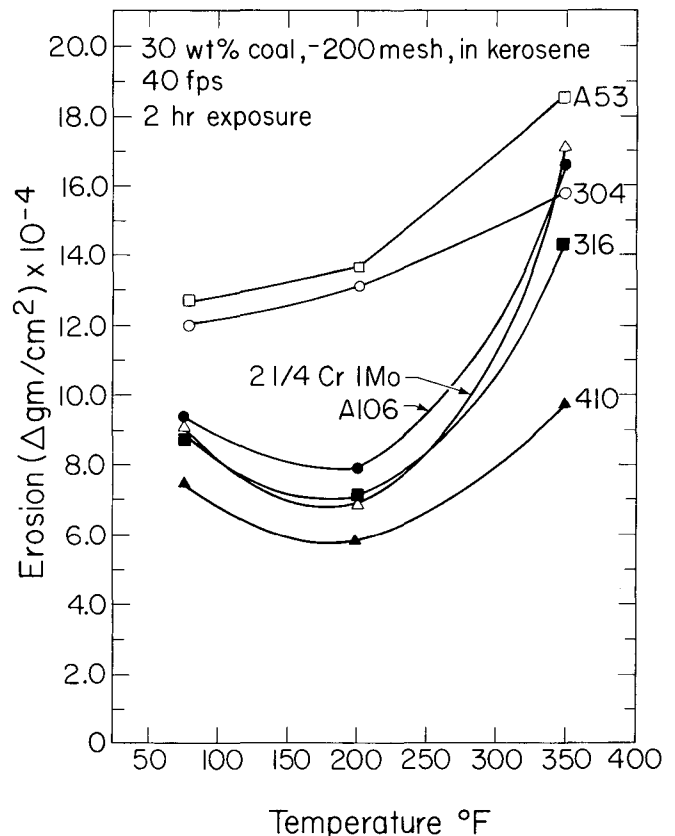


Fig. 1. Effect of test temperature on the erosion of alloys by a coal-kerosene slurry flow.
(XBL 8110-7151A)

*This work was supported by the Assistant Secretary for Fossil Energy, Office of Coal Research, Materials Components Division of the U. S. Department of Energy under Contract No. DE-AC03-76SF00098 through the Fossil Energy Materials Programs, Oak Ridge National Laboratory, Oak Ridge, TN.

steels. The increase in erosion rate at the 177°C test temperature is primarily due to the decrease in the viscosity of the kerosene, which increases the erosivity of the slurry.

In other tests using hexadecane with and without hexadecanoic acid as the carrier liquid for the pulverized coal, it was determined that the lubricity of the liquid plays a major role in the erosivity of slurries. The liquid containing the 1/2 percent acid had a higher lubricity at the same viscosity, which resulted in a major reduction in the amount of erosion on the A53 mild steel and 304SS.

* * *

†Brief version of LBL-13804.

2. THE EROSION MECHANISM OF STEELS IN COAL-KEROSENE SLURRIES[†]

Phil Tom and Alan Levy

The mechanism of erosion of several alloy steels was investigated. An understanding of the active mechanism as it is affected by the test variables in a slurry environment is required for a predictive model of erosion. The selection and design of improved alloys for use in slurry-containing components of coal conversion and combustion system components also requires an understanding of the active mechanism of erosion. The knowledge of erosion mechanisms of ductile alloys which was developed in a complimentary gas-solid particle erosion study was used in this project.

A jet impingement tester capable of directing a stream of coal-kerosene at a flat test specimen was used. Eighteen gallons of slurry flowing at velocities ranging from 100 to 200 m/s were suf-

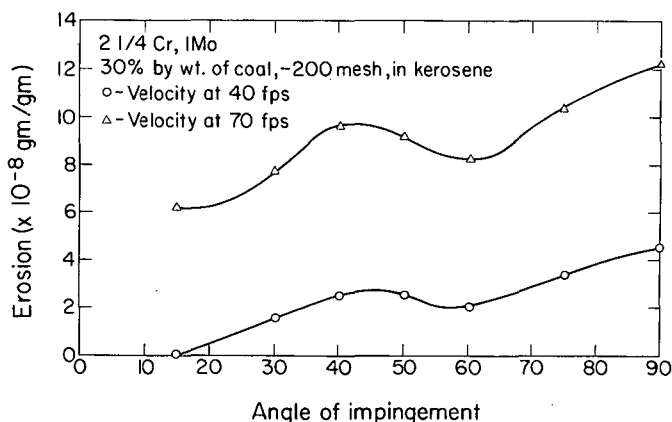


Fig. 1. Effect of angle of impingement of the erosion of 2-1/4Cr1Mo steel in a coal-kerosene slurry flow. (XBL 8110-7150A)

ficient to produce reliable weight losses of the 2.5 x 3.5 cm test specimens at 25°C.

The effect of the impingement angle of the eroding stream on the erosion rate of an alloy steel, 2-1/4Cr1Mo, is shown in Fig. 1. An initial erosion peak occurs at an angle of 40°. A peak at 20° occurs in gas-solid particle erosion. The increased angle of the peak for the coal slurry is the result of the increased viscosity and lubricity of the slurry compared to a gas-solid particle stream. The amount of erosion in a gas-solid particle stream is roughly 4 orders of magnitude greater than that which occurs in a nonaqueous liquid-solid particle stream at the same particle velocity. The increase in erosion rate to the 90° impingement angle also differs from that in gas-solid particle streams, which cause lower erosion rates to occur after 20°. Lubricating liquids cause increased erosivity of the particles at steeper angles because the liquids carry particle kinetic energy to the eroding surface more effectively.

* * *

†Brief version of LBL-13805.

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Papers Presented

1. A. Levy, "Erosion of Metals in Nonaqueous Coal Slurries," NACE, S.F. Chapter Meeting, Berkeley, CA, April 14, 1981.
2. A. Levy and G. Hickey, "Slurry Erosion Behavior of Alloy Steels at Elevated Temperatures," AIME-TMS Meeting, Louisville, KY, October 15, 1981.
3. A. Levy and P. Tom, "Jet Impingement Slurry Erosion of Alloy Steels," AIME-TMS Meeting, Louisville, KY, October 15, 1981.

d. Utilization of Metals in Oil Shale Retort Components*

Alan V. Levy, Investigator

Introduction. Work on the corrosion of low alloy steels and 300 series stainless steels in the high temperature oxidizing and sulfidizing environments of in-situ oil shale retorts was concluded and attention was turned to corrosion in above-ground retorts. An above-ground retort has the same corrosive sulfur and oxygen containing environment, but the lower operational temperatures do not impose such a severe corrosion environment on metals. Corrosion behavior of steels in retorted shale oil was also investigated.

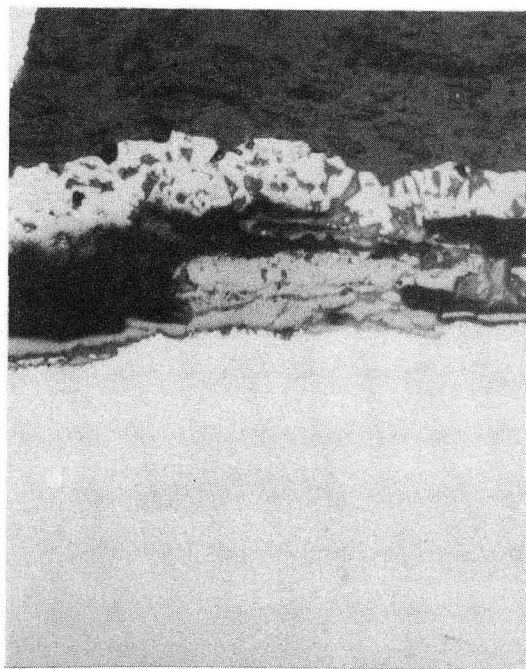
1. HIGH TEMPERATURE CORROSION

Elliott Slamovich and Alan Levy†

The corrosion of low-alloy and stainless steels was determined in above-ground, simulated in-situ oil shale retorts by exposing 2.5 cm x 7.5 cm x 6 mm specimens in the 10-ton experimental retort at the Laramie Energy Technology Center (LETC). Further corrosion studies were conducted in a labora-

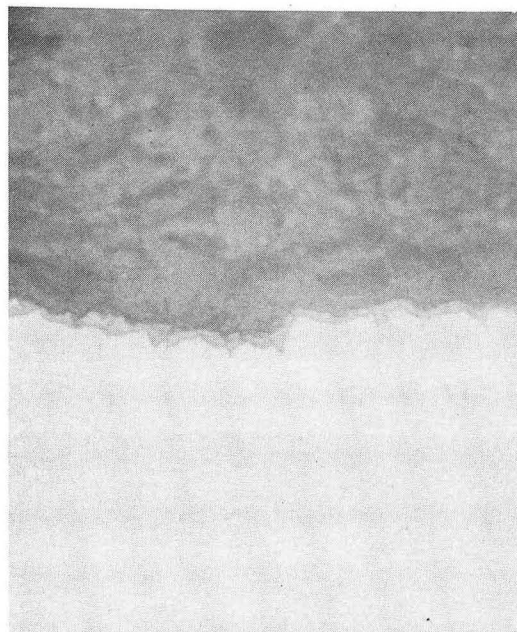
tory furnace crucible containing 15 grams of oil shale to determine the oxidation and sulfidation behavior of different alloys in a more controllable environment than occurs in the LETC retort. Sections of thermowell piping in underground in-situ retorts were also examined to study the effects of long term exposure to the retorting environment.

Test samples were run at LETC exposed to Moroccan "M" Zone high sulfur (4-5%) shale. The retorting run lasted only 30 hours and the temperature spiked at 1100°C, staying above 815°C for only six hours. Figure 1 shows the scales formed on 1018 plain carbon and 304 stainless steel. The difference in chromium content between the two steels, 0% and 18% respectively, was the main factor in the difference in the corrosion behavior observed. The 1018 steel has an outer layer of iron with deposits of iron oxide in it and an inner layer of iron oxide - iron sulfide directly attached to the remaining metal. The stainless steel has a thick layer of protective chromium



1018 Carbon Steel

25 μ m



304 Stainless Steel

25 μ m

Fig. 1. Corrosion scales on 1018 steel and 304SS.

(XBB 8112-11950)

*This work was supported by the Assistant Secretary for Fossil Energy, Office of Coal Research, Materials Components Division of the U. S. Department of Energy under Contract No. DE-AC03-76SF00098.

oxide on its surface with little or no attack down the grain boundaries. More extensive attack of both alloys would have occurred if the retorting operation had been longer.

De-emphasis of in-situ oil shale retort research has prevented LETC from testing the 2-1.4%, 5%, 9% and 12% chromium content steels. These alloys and various other low alloy and stainless steels are being tested in a laboratory crucible containing unreacted Anvil Pts. shale (1.7% S, 24 gal/ton) at 1000°C for 48 hours.

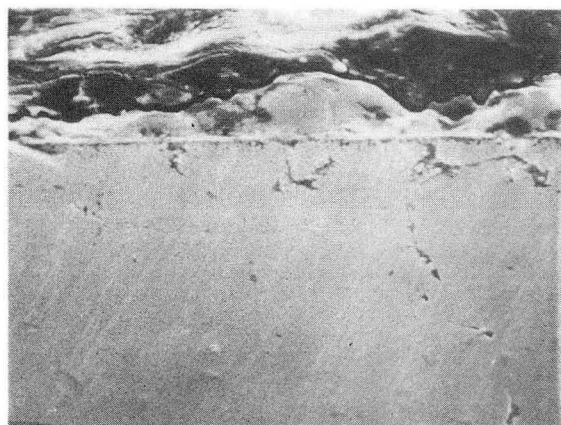
Figure 2 shows 304SS after a 48 hour exposure to shale. The outer scale is primarily chromium sulfide. A thin chromium oxide layer is between the outer scale and the metal. Internal sulfidation is found primarily along grain boundaries resulting from the inability of the chromium oxide layer to act as a diffusion barrier to sulfur. Diffusion of sulfur through the Cr₂O₃ protective scale duplicates the behavior of austenitic stainless steels in simulated in-situ retort exposures at LETC. The ability of the laboratory crucible to simulate the corrosion conditions that occur in large scale retorts was demonstrated. Analysis of

exposures of other chromium containing steels showed a direct relation between corrosion resistance in an in-situ oil shale report and the chromium content of the alloy. Alloys with 12-18% Cr exhibited much higher corrosion resistance than alloys with 2 1/4%-9% Cr.

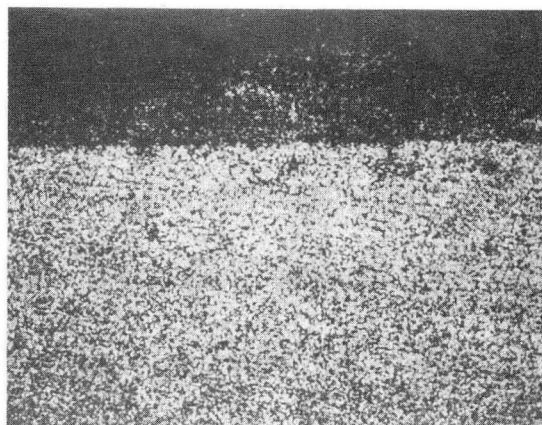
Analysis of a plain carbon steel pipe used as a thermowell protector in an actual underground in-situ retort (Fig. 3) shows a high level of corrosion with the formation of a porous layered scale on the base metal shown on the right side. X-ray maps show that there are three layers: a layer next to the base metal of iron oxide with a small amount of sulfur in it, a middle layer of iron sulfide, and an outer layer of iron oxide. At other locations along the 0.25-in. thick pipe, the metal was completely consumed, leaving layers of iron oxide and iron sulfide. The severe attack is typical of that of nonchromium containing alloys in in-situ oil shale retorts.

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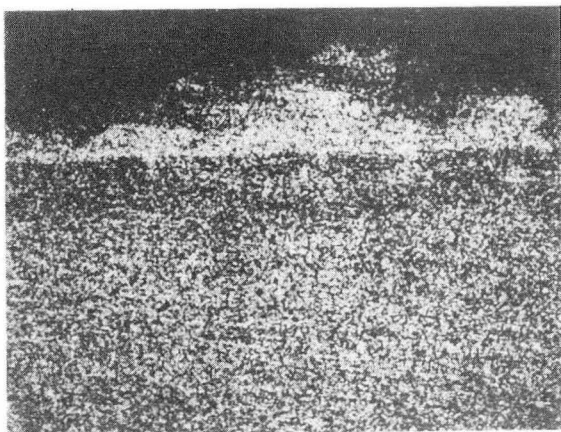
†Brief version of LBL-13809.



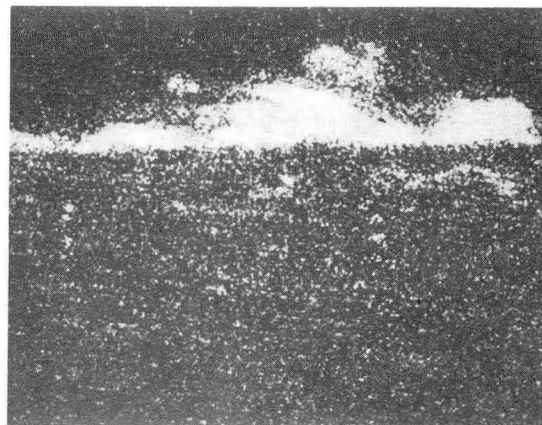
10_μm



Fe Map



Cr Map



S Map

304SS, 48hrs., 1000°C

Fig. 2. Corrosion scale formed on 304SS in laboratory crucible test.
(XBB 8112-11952)

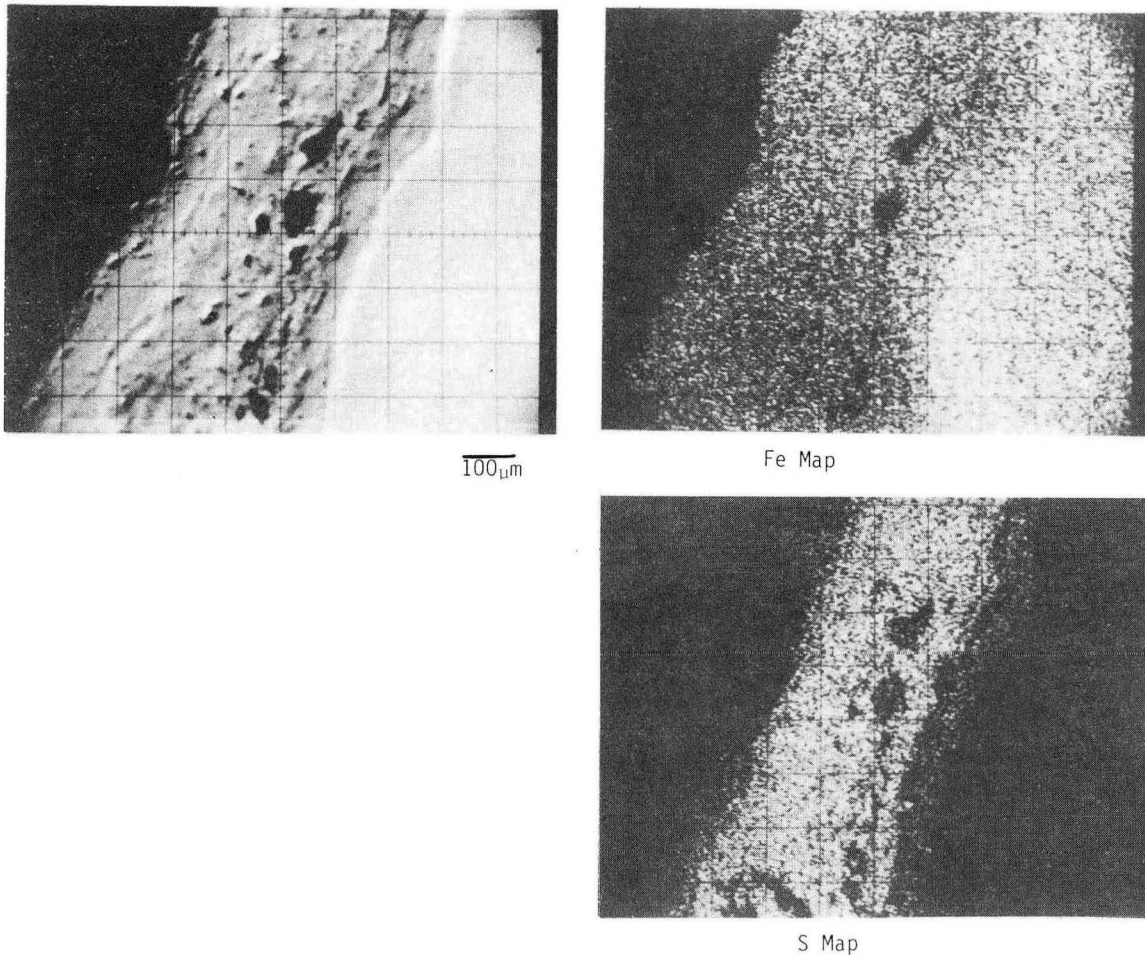


Fig. 3. Corrosion scale on a plain carbon steel pipe from an in-situ retort. (XBB 8112-11951)

2. LOW TEMPERATURE CORROSION

Elliott Slamovich, Jennifer Okano, and Alan Levy

As in the high temperature studies, tests were conducted in the field and laboratory. Samples 2.5 cm x 7.5 cm x 6 mm were exposed in a large scale, above ground retort in Colorado at temperatures to 500°C for 400 hours. Other exposures were made in LBL's 7.5-cm diam retort at 550°C for 48 hours. The effect of partial and total immersion in shale oil on the corrosion of steel was also studied at temperatures to 300°C.

Tests in the large scale, above ground retort showed mild corrosion only on the 1018 carbon steel. No corrosion was observed on the stainless steels exposed. Tests in the LBL 7.5-cm diam retort at 550°C showed mild attack only on the plain carbon 1018 steel. Exposure to shale oil at 300°C for 100 hours showed essentially no corrosion on

stainless steels and only mild corrosion on 1018 steel. These results indicated that there will probably be little or no corrosion problems in above ground oil shale retort operations.

1981 PUBLICATIONS AND REPORTS

Other Publications

1. D. Whittle, M. El Dahshan, and A. Levy, "Corrosion of Materials in In-Situ Oil Shale Environments," Proc. 8th International Congress on Metallic Corrosion, ICC, Mainz, West Germany, September 6-11, 1981.

LBL Report

1. E. Slamovich, J. Okano, and A. Levy, "Corrosion of Metals in Oil Shale Retorting Environments," LBL-13809.

Presentations

1. A. Levy, W. Goward, and D. Boone, "High Temperature Corrosion of Metals and Coatings in Oil Shale Environments," 4th Annual Oil Shale Conversion Symposium, Denver, CO, March 25, 1981.

2. A. Levy and R. Bellman Jr., "Corrosion of

Metals in Oil Shale Environments," NACE Corrosion 82, Toronto, Ont., April 6, 1981.

3. A. Levy, "Corrosion of Metals in Oil Shale Retorting Environments," ASM Spring Seminar, San Diego, CA, April 16, 1981.

h. Materials Characterization in Fossil Fuel Combustion Products*

Donald H. Boone, David P. Whittle, and Alan V. Levy,
Investigators

Introduction. The durability of directly fired heat engines operated on coal-derived liquids, shale oil, coal slurries, and other minimally processed alternate fuels is dependent on the surface stability of combustion zone components. Some type of protective coating system will be mandatory if the potential advantages of lower fuel costs and more efficient combustion are to be fully realized.

The DOE Combustion Zone Durability Program is involved in the development and evaluation and the materials technology necessary for the successful use of these fuels.

The LBL portion of this effort is focused on developing a better understanding of the structure of the various types and forms of ceramic thermal barrier coatings and their behavior in a variety of testing and engine environments. Additional LBL functions in this effort include the coordination and documentation of test specimen production, testing and analysis for the entire program, and participation on the Combustion Zone Durability Steering Panel.

1. PLASMA SPRAYED THERMAL BARRIER COATINGS[†]

Donald H. Boone

Ceramic coatings are currently being evaluated for a number of thermal barrier applications because the coatings provide both insulation, allowing hotter gas temperatures, and an inert barrier to isolate the metallic components from the corrosive environment. Plasma sprayed ZrO_2 is a prime candidate, and this study is concentrating on determining the effects of stabilizing MgO or Y_2O_3 additions in modifying the protective properties of the coating.

The initial plasma sprayed ZrO_2 studies involve structures prepared by the Central Institute of Industrial Research (CIIR), Oslo, Norway.

*This work was supported by the Assistant Secretary for Fossil Energy, Office of Coal Research, Heat Engines and Heat Recovery Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098 through the Battelle Memorial Institute, Pacific Northwest Laboratories, Richland, WA.

These ceramic coatings are undergoing tests in slow speed ship diesel engines. Preliminary results show a benefit of the MgO-stabilized ZrO_2 over the Y_2O_3 -stabilized ceramic (YSZ). Similar structures prepared by CIIR and tested in a Trans-america Delaval diesel on exhaust valves showed an apparent reversal of these results after a 4000 hour exposure. In other test programs, it has been found that the MgO-stabilized ZrO_2 is more resistant to vanadium-induced attack. The cleaner fuels used by Delaval in their tests may reflect a measure of resistance to thermally induced stresses rather than to chemical attack.

* * *

[†]Brief version of LBL-13777.

2. THERMAL BARRIER COATINGS APPLIED BY PHYSICAL VAPOR DEPOSITION[†]

Donald H. Boone

Efforts to improve the thermal fatigue resistance of ceramic thermal barrier coatings by structural modification include segmentation by chemical and mechanical means. A promising approach is the use of the characteristically columnar oriented growth structure produced by physical vapor deposition (PVD) such as electron beam evaporation and sputtering. This study, in collaboration with Airco-Temescal, is concentrating on the effects of the deposition variables on the structure and adherence of electron beam PVD applied Y_2O_3 - ZrO_2 .

The oriented growth structure of the PVD-deposited ceramic ensures a low effective modulus in a direction perpendicular to the columnar grains and, thus, any thermally induced strains are only weakly transmitted to the ceramic-metal interface, resulting in improved thermal fatigue behavior. Processing variables such as deposition temperature and rate, initial surface condition and geometry all affect the development of the columnar structure. Figure 1 shows a fracture section through a PVD fully stabilized ZrO_2 coating deposited at about 800°C. The high length/diameter ratio of the columnar grains and the sensitivity of the ceramic-metal interface to adherence (lower figure) are clearly seen. Increase in the deposition temperature of 600°C to 1100°C resulted in an increase in average columnar diameter. Surface temperature cycling with rotation during deposition resulted in detrimental renucleation

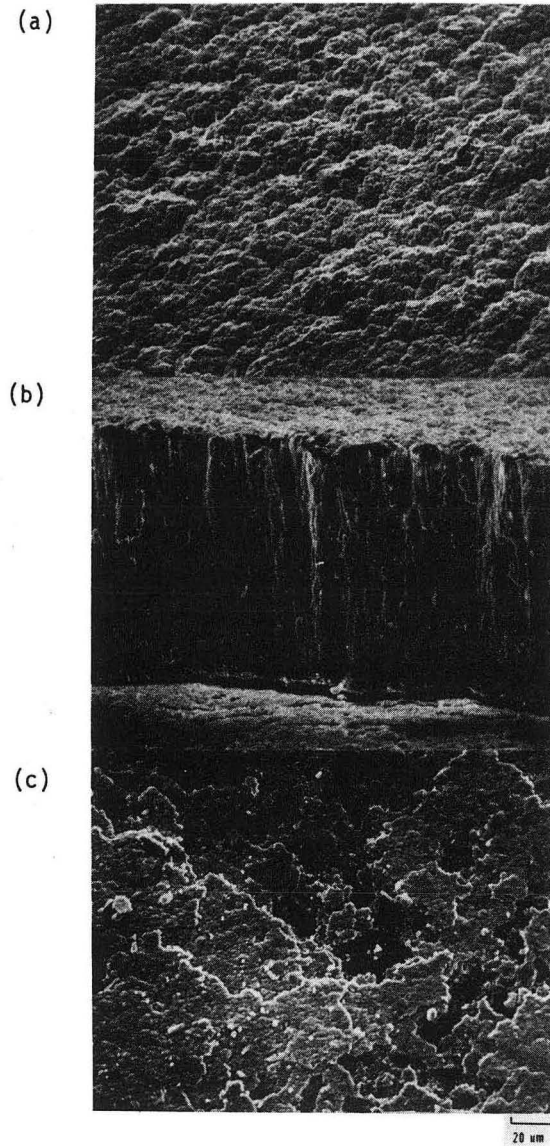


Fig. 1. Microstructure of an electron beam physical vapor deposited Y_2O_3 -stabilized ZrO_2 coating: (a) surface, (b) fracture cross-section, and (c) interface surface between deposited ZrO_2 and metallic interlayer.

(XBB 817-5936)

and interrupted column growth.

Future studies will concentrate on ceramic-metal or ceramic-oxide-metal coating systems since the substrate surface is often a pre-oxidized interface.

* * *

† Brief version of LBL-13721.

3. INTERACTIONS BETWEEN COATINGS AND SUBSTRATES AT ELEVATED TEMPERATURES[†]

Donald H. Boone and David P. Whittle

Three principal types of coatings have been developed: diffusion aluminides, overlay coatings, and ceramic or thermal barrier coatings. This order of development also represents what might at first be anticipated to be an increasing independence of the substrate. However, it is slowly being realized that this is certainly not the case for aluminide and overlay coatings, and is probably not even true for ceramic coatings.

An important question is which are the beneficial and detrimental substrate elements incorporated into the coating and to what extent do they interact with protective oxide formation on the coating. W, Mo, and Ta may contribute toward acid fluxing of the oxide in service conditions involv-

ing sulfate contamination; Ti, Hf and Zr may contribute to scale adhesion or compete with Al for protective oxide formation. Calculations have shown that at temperatures above about 1000°C, substrate elements can diffuse rapidly through a nominally 150 μm thick coating. However, at lower temperatures, effects of the substrate elements were still present in the coatings, even at short exposure times, which strongly suggests that some elements are able to diffuse rapidly during the coating deposition process itself. This hinders the coating from developing a truly protective Al₂O₃ film.

* * *

†Brief version of LBL-13350.

1981 PUBLICATIONS AND REPORTS

Refereed Journals

1. J. Maasberg, D. H. Boone, D. P. Whittle, and A. V. Levy, "Erosion Resistance of CoCrAl Coatings Containing Active Element Additions," *Thin Solid Films* 84, 315-322 (1981); LBL-12016.
2. D. H. Boone, D. A. Crane, and D. P. Whittle, "The Structure and Interdiffusional Degradation of Aluminide Coatings on Oxide Dispersion Strengthened Alloys," *Thin Solid Films* 84 (1981).

Other Publications

1. D. P. Whittle and D. H. Boone, "Oxidation Characteristics of CoCrAl Type Protective Coatings," *Proc. 8th Int. Congress on Metallic Corrosion, Vol. I, Frankfurt, 1981; publ. by DECHEMA (1981) 718-723.*
2. D. H. Boone and M. E. Browning, "The Advancing Deposition Process," *Proc. of 68th Annual AES Meeting, Boston, MA. (1981).*
3. D. P. Whittle and D. H. Boone, "Interactions Between Coatings and Substrates at Elevated Temperatures," *Proc. of the DOE-EPRI Second Conf. on Advanced Materials for Alternative-Fuel Capable Heat Engines, Monterey, CA. (Aug. 1981); LBL-13350.*

LBL Reports

1. E. Demeray, J. W. Fairbanks, and D. H. Boone, "Physical Vapor Deposition of Ceramic Coatings for Gas Turbine Engines," LBL-13721.
2. D. A. Crane, R. Lambertson and D. H. Boone, "The Protective Oxide Characterization of Mn Modified Electrophoretically Applied Aluminide Coatings," LBL-13776.
3. D. A. Crane, R. Lambertson and D. H. Boone,

"Characterization of Plasma Spray Applied Ceramic Coated Diesel Engine Exhaust Valves. Part I As-coated Condition and Part II Engine Tested," LBL-13777.

Invited Talks

1. D. H. Boone, "Mechanical Property Effects of Protective Coatings for Energy Conversion Systems," Golden Gate Metals and Welding Conf., San Francisco, CA., January 1981.
2. D. P. Whittle, D. H. Boone and S. Shaffer, "Oxidation Properties of CoCrAl-based Coatings," AIME Annual Meeting, Chicago, IL., February 1981.
3. D. H. Boone, D. P. Whittle and D. A. Crane, "The Structure and Interdiffusional Degradation of Aluminide Coatings on Oxide Dispersion Strengthened Alloys," AIME Annual Meeting, Chicago, IL., February 1981.
4. D. H. Boone, W. D. Goward and P. G. Moore, "Structure of Laser Treated MCrAlY Coatings," Int. Conf. on Metallurgical Coatings, San Francisco, CA., April 1981.
5. D. H. Boone, D. A. Crane and D. P. Whittle, "Oxide Structure on Aluminide Coatings," Int. Conf. on Metallurgical Coatings, San Francisco, CA., April 1981.
6. D. P. Whittle, S. Shaffer and D. H. Boone, "Relationships Between Oxide Morphology and Coating Structure," Int. Conf. on Metallurgical Coatings, San Francisco, CA., April 1981.
7. D. H. Boone, "Films and Coatings for Technology," Continuing Education Institute Short Course, Los Angeles, CA., May 1981.
8. D. H. Boone, "Selection and Processing of High Temperature Coatings, Deposition Technologies and Applications Short Course, Linkoping University, Sweden, June 1981.
9. D. H. Boone, "Coatings for Gas Turbines," IPAT-81 (Ion Plating Assisted Technologies), Amsterdam, Netherlands, June 1981.
10. D. H. Boone, "Recent Advances In Coating Deposition Techniques," Linkoping University, Sweden, November 1981.
11. D. H. Boone, "Substrate Effects on Coating Protective Oxide Formation," Brown, Boveri and Cie, Central Research Institute, Heidelberg, West Germany, November 1981.
12. D. H. Boone, "The Science of Protective Coatings," MTU Motoren und Turbinen-Union, Munchen, West Germany, November 1981.

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