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An Evaluation of 2¼Cr-1Mo Steel for Liquid-Lithium Containment.

III. Effect of Velocity and Lead Content

**Unabridged Final Report
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An Evaluation of 2-14Cr-1Mo Steel
for Liquid Lithium Containment
III. Effect of Velocity and Lead Content

(Contract No. 8484909)

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FORWARD

This report is the final unabridged report covering research performed under Contract No. 8484909 of the Laser Fusion Program, and was administrated by Lawrence Livermore National Laboratory under the supervision of Mr. J. Hovingh (LLL) and Dr. N. J. Hoffman of the Energy Technology Engineering Center. The research was performed by B. D. Wilkinson under the direction of Professor G. R. Edwards.

ABSTRACT

The intergranular penetration of specially heat treated 2-1/4Cr-1Mo steel by lead-lithium liquids containing 0, 17.6, and 53 w/o lead has been investigated at temperatures ranging from 300°C to 600°C and for times to 1000 hours. Limited tests using a 99.3 w/o lead-lithium liquid were also conducted.

The corrosion reaction observed, when 2-1/4Cr-1Mo steel is corroded by 0, 17.6, and 53 w/o lead-lithium liquids, followed parabolic rate dependency. Two rates of penetration were observed during steel exposure to the liquids, an initial rapid penetration rate followed by a slower secondary penetration rate. The difference observed between the two rates of penetration is believed to be associated with the thermodynamic stability, with respect to lithium, of the carbides present.

Tempering was found to remove the susceptibility of specially heat treated 2-1/4Cr-1Mo steel to penetration at 500°C by lead-lithium liquids containing up to 53 w/o lead. In addition, penetration by a 99.3 w/o lead-lithium liquid

in 1000 hours and at 500°C was found to be negligible even when the steel was in the most susceptible condition.

During this investigation it was determined that previous Arrhenius analysis of other lead-lithium corrosion data on 2-1/4Cr-1Mo steel yielded an erroneously high activation energy for long exposure times. By using a corrected analysis it was determined that the long exposure time activation energy was approximately equal to that obtained for short exposure times. Because of this reanalysis, the penetration of 2-1/4Cr-1Mo steel by a 17.6 w/o lead-lithium liquid is now explained as controlled by the diffusion of carbon in the liquid over all exposure times.

Arrhenius analyses of 2-1/4Cr-1Mo steel steel corroded by 0 and 53 w/o lead-lithium liquids yielded similar activation energies. Therefore, it was concluded that the corrosion mechanism by which 2-1/4Cr-1Mo steel is penetrated does not vary for lead concentrations in the lithium up to 53 w/o lead.

From this investigation it was confirmed that an anomaly exists at 500°C for the corrosion of 2-1/4Cr-1Mo

steel by lead-lithium liquids containing from 0 to 53 w/o lead. This behavior is hypothetically explained by the existence of competitive processes. These processes are the formation and dissolution of a corrosion product at the corrosion front.

A limited number of simple dynamic tests were performed utilizing a spinning disk technique. Results from this testing showed that the penetration of 2-1/4Cr-1Mo steel by a 17.6 w/o lead-lithium liquid at 500°C was not sensitive to angular velocities up to 4.46 revolutions per second.

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INTRODUCTION

1.1 General Background

An extremely viable concept for power generation is the inertial confinement fusion reactor. This concept utilizes controlled laser fusion for energy generation. In addition, a thick lithium blanket will be used to provide significant protection from neutron radiation damage in the first structural wall, act as the reactor coolant, and create fuel through neutron conversion of lithium to tritium. However, one major drawback to the use of lithium is that many structural alloys are severely corroded by liquid lithium. Liquid lithium corrosion susceptibility therefore is one of the major alloy selection criteria in inertial confinement designs.

Engineering alloys for lithium containment in these designs must meet criteria of availability, fabricability, and code acceptance, in addition to having acceptable resistant to liquid lithium corrosion. The Hylife plant concept (1), one such design, utilizes 2-1/4Cr-1Mo steel as

the potential candidate for the confinement of the liquid blanket. This design might also employ a liquid lead-lithium alloy as the blanket material. The Hylife design has therefore created an interest in investigating and characterizing the effect of lead in liquid lithium corrosion of 2-1/4Cr-1Mo steel.

1.2 Purpose

The corrosive nature of liquid lithium is well known (1-25); however, very little is known about the corrosion behavior of lead-lithium liquids. This study's purpose was to investigate and characterize the effect of lead concentration on the corrosion phenomena observed when regular grade 2-1/4Cr-1Mo steel was exposed to lead-lithium liquids at elevated temperatures.

In developing a clear understanding of the corrosion behavior of lead-lithium liquids, background information concerning previous lithium and lead corrosion studies, and the physical metallurgy and relative stability of the steel phases to lithium were important concepts to consider.

1.3 Previous Corrosion Data

Corrosion of various alloys by lithium has been studied by several investigators (1-25). Initial work (2-14) on the general corrosion of materials by molten lithium did establish a general ranking of materials with regard to this susceptibility, but failed to recognize the importance of impurity elements and dissimilar-metal transfer effects. Later studies (10,19,20) revealed that the lithium corrosion of stainless steel was very sensitive to the nitrogen concentration in either the metal or the lithium. Nitrogen was also found to be of importance in the corrosion rate observed in 2-1/4Cr-1Mo steel when exposed to liquid lithium (20). In a different study (1), it was shown that the heat affected zones of 2-1/4Cr-1Mo steel weldments were very susceptible to penetration by the liquid lithium, while the tempered parent metal was quite resistant to attack.

Studies of the effect of lead on metals have been no more extensive than the lithium corrosion studies. Most research has concentrated on the lead embrittlement of steels (26-28), but limited studies of the general corrosion of steels by lead have shown significant effects (29,30).

Romano, et.al. (29) found that 2-1/4Cr-1Mo steel was susceptible to attack by lead and lead alloys. Later work by Asher, et.al. (30) reconfirmed the early study and found that lead attack of 2-1/4Cr-1Mo steel could be reduced or eliminated by adding titanium in ppm levels. Asher also found that the attack by molten lead on structural materials was related to: 1) the solubility of the specific alloying elements in the lead, and 2) whether or not there was a protective film (i.e. oxide) present on the material. Table I gives various solubilities of elements of interest in liquid lead at 700°C.

TABLE I

Solubilities of Various Elements
in Liquid Lead at 700°C (30)

Element	Solubility(ppm)
Iron	8.0
Chromium	6.0
Molybdenum	0.1

The corrosion of 2-1/4Cr-1Mo steel by a 17.6 w/o lead-lithium liquid was studied by Anderson (24), but he did not address the effect lead may have on the corrosion

phenomena observed. He exposed GTA weld coupons of both regular and niobium-stabilized grades of 2-1/4Cr-1Mo steel to a 17.6 w/o lead-lithium liquid at a temperature of 500°C and found that the as-quenched bainitic microstructure, (a microstructure similar to the heat affected zone of a weld), was readily penetrated at the prior austenite grain boundaries. Results also showed that with a proper post-weld heat treatment, austenite grain boundary attack could be greatly reduced, and that niobium-stabilized material exhibited very good corrosion resistance even in the as-welded condition. Anderson heat treated coupons to produce microstructures similar to the most susceptible microstructure of a weldment, (the heat affected zone right next to the fusion line). He then exposed them to liquid 17.6 w/o lead-lithium at temperatures ranging from 400°C to 625°C, and found that the corrosion kinetics for penetration followed a parabolic rate dependence. Two rates of penetration occurred, depending on the time and temperature.

Figure 1 presents the results Anderson (24) obtained from his penetration experiments, and Figure 2 shows his Arrhenius plot used in determining the apparent activation energies for the two distinct rates of penetration. The

Figure 1. Corrosion of simulated weldments (oil quenched from 1300°C) as a function of time^{1/2} and temperature (24).

Figure 2. Arrhenius plot of the corrosion rate constants taken from Figure 1 (24).

first mechanism, having an activation energy around 22 kJ/mole and believed to be liquid diffusion-controlled, correlated with the predicted existence of epsilon carbide and its dissolution by lithium. The latter mechanism, with an activation energy of 118 kJ/mole and believed to be reaction controlled, seemed to be associated with the dissolution of cementite by the lithium. By utilizing the work of Baker and Nutting (31) for the carbide transformations in 2-1/4Cr-1Mo steel during tempering, Anderson showed that the time of transition between the initial and secondary mechanisms for a given corrosion test approximately corresponded to the time required for transformation of epsilon-carbide to cementite. Figure 3 shows these results.

Figure 3. Comparison of the change in the corrosion rate with the conversion of epsilon carbide to cementite in quenched 2-1/4Cr-1Mo steel (24).

1.4 The Physical Metallurgy of 2-1/4Cr-1Mo Steel

Strength, creep resistance, oxidation resistance, corrosion resistance, and service lifetime requirements were the major factors influencing the original development of 2-1/4Cr-1Mo steel. The alloy was developed for the power generation industry, which has successfully used the steel in heavy section and boiler tube applications for many years. In addition, vast amounts of research and development have been done on the steel with respect to fabrication, joining technology, and improvement of mechanical properties.

Figure 4 is the continuous cooling transformation diagram for 2-1/4Cr-1Mo steel (32). The diagram shows a steel possessing a low hardenability with respect to martensite formation, but also shows a steel which has extreme difficulty in transforming to pearlite. These transformation characteristics are somewhat unique among carbon and low alloy steels. The final microstructure upon cooling tends to be completely bainitic or a combination of proeutectoid ferrite and bainite, depending on cooling rate. Commercially the steel is supplied in one of three conditions; annealed, normalized, or quenched. The

Figure 4. Continuous cooling transformation diagram for 2-1/4Cr-1Mo steel: F=polygonal ferrite; P=pearlite; B=bainite; M=martensite (32).

microstructural differences in the final condition are determined by the cooling rate to which the steel is subjected after austenitizing. The cooling rate affects the relative amounts of ferrite and bainite present in each condition.

Baker and Nutting (31) performed tempering experiments on 2-1/4Cr-1Mo steel which possessed two different cooling histories after austenitizing (normalized and quenched), and observed the carbide transformations as a function of temperature and time. Figures 5a and b are the results from their experiments. They determined that, although the overall sequence of carbide transformation for each initial condition was similar, significant differences, associated with the differences in the initial microstructures, could be observed. The carbide sequences for the two cooling rates upon tempering can be summarized as follows:

Normalized Condition:

(i) bainite

epsilon carbide		cementite -----	Cr_7C_3
+		+	
cementite	--- cementite ---	Mo_2C ---	M_{23}C_6 --- M_6C

a) QUENCHED

b) NORMALIZED

Figure 5. Carbide stability diagrams for a) quenched and tempered and b) normalized and tempered 2-1/4Cr-1Mo steel (31).

(ii) ferrite

Quenched Condition:

These carbide sequences reflect the relative thermodynamic stability of the various carbides and morphologies.

The stability of the various phases and morphologies in 2-1/4Cr-1Mo steel during exposure to liquid lithium is important. Previous data (1,24) have shown that the ferrite microconstituent is quite resistant to attack by liquid lithium and that the dissolution of epsilon carbide and cementite within the bainite is responsible for penetration. Figure 6 (33) is a plot of the free energy of formation versus temperature curves for various carbides exposed to carbon saturated liquid lithium. Although epsilon carbide is not shown on the plot it is known to be less stable than cementite at all temperatures in this steel (31). Epsilon carbide, cementite, and Mo₇C are all unstable with respect to

Figure 6. Relative thermodynamic stability of several carbides in carbon saturated lithium (33).

Li_2C_2 at the design temperature of 500°C . In fact, cementite is less stable than Li_2C_2 up to 650°C , while Mo_2C is less stable up to about 623°C . From these thermodynamic data, it can be seen that less stable carbide phases should dissolved in the presence of carbon saturated lithium.

1.5 Experimental Scope

In addition to the effect of lead, numerous other parameters, all potentially deleterious to the lithium corrosion resistance of 2-1/4Cr-1Mo steel, remain to be investigated. One uninvestigated variable is the effect of velocity on the corrosion behavior of lead-lithium liquids. Preliminary studies of the effects of lead content and velocity on the corrosion susceptibility of regular grade 2-1/4Cr-1Mo steel were therefore conducted.

1.5.1 Microstructures Studied

Because plates of 2-1/4Cr-1Mo steel must be joined by a welding process to form the containment vessel, various microstructural differences will exist in the steel during service. These include the microstructures of the fusion, heat affected, and base metal zones. Anderson (24) has demonstrated that the most susceptible region of a weld to liquid lithium attack is the heat-affected zone, and that the heat affected zone microstructure can be simulated by quenching the steel from a high austenitizing temperature. Specimens of 2-1/4Cr-1Mo steel, in this study, were

consequently given the heat treatment appropriate to simulate the corrosion susceptible HAZ microstructure.

1.5.2 Static Liquid Tests

Coupons susceptible to lithium attack were exposed to different lead-lithium liquids, as well as to pure lithium, at temperatures from 300°C to 600°C. The specific lead concentrations in weight percent were 0, 17.6, 53, and 99.3 w/o. Additional coupons were given a post weld heat treatment prior to lithium exposure for the purpose of evaluating the effectiveness of the heat treatment in preventing penetration by the liquids. For comparative purposes, selected GTA spot welds were also exposed to some of the liquids.

After penetrations were determined on each coupon, corrosion rate values were obtained for the two distinct penetrations according to a penetration squared versus time equation. These corrosion rate values were then analyzed according to an Arrhenius analysis for apparent energy values. From the activation energies obtained, efforts were made to support believed rate controlling mechanisms for the observed corrosion of 2-1/4Cr-1Mo steel exposed to liquid

lead-lithium alloys.

1.5.3 Dynamic Liquid Tests

In addition to static corrosion tests, simple dynamic tests were conducted in a spinning disk apparatus. The simulated HAZ microstructures, both untempered and post weld heat treated, were examined at three velocities (0, 2.23 ω , and 4.46 ω) in a 17.6 w/o lead-lithium liquid at 500°C.

EXPERIMENTAL PROCEDURE

2.1 General

Table II lists the chemical compositions of the materials used during this investigation of static and dynamic liquid lead-lithium alloy corrosion.

TABLE II

Chemical Composition of Test Materials (w/o)

TEST	C	Mn	Si	Cr	Mo	P	S	Fe
STATIC	0.13	0.23	0.44	2.25	0.97	0.014	0.022	bal.
DYNAAMIC	0.12	0.43	0.32	2.07	1.04	0.018	0.002	bal.

All material was given an initial isothermal transformation heat treatment that consisted of austenitizing at 927°C, cooling 50°C/hour to 704°C, holding for two hours, cooling 50°C/hour to 500°C, and furnace cooling to room temperature. The heat treatment was conducted in a vacuum furnace (10^{-4} torr) and was chosen to

approximate the microstructural conditions present in the material before welding. This heat treatment is similar to that used by Klueh, et. al. (34-39) to enhance the creep resistance of 2-1/4Cr-1Mo steel at elevated temperature. Figure 7 is a micrograph of the structure obtained after this heat treatment. The microstructure shown consists of proeutectoid ferrite (light areas) with about 20 percent bainite (dark areas). In addition, it has been reported (39) that small amounts ($\approx$ 1 percent) of pearlite may be present within the bainite regions. The carbide phases present after this isothermal heat treatment were found to be M_3C and Mo_2C (39). In the proeutectoid ferrite regions only Mo_2C has been observed, while both carbide phases have been seen in the bainite regions.

Once the isothermal transformation heat treatment was completed, the material was sectioned and/or machined before given a heat treatment to simulate the heat affected zone (HAZ) of a weldment. This was accomplished by austenitizing at $1300^{\circ}C$ ($\approx$ ten minutes) and oil quenching. The heat treatment was performed with the aid of a glo-bar furnace in normal atmospheric conditions (79% N and 21% O). A portion of this material was given a post-weld heat treatment or temper of ten hours at $760^{\circ}C$. Figures 8a and b are

Figure 7. Microstructural appearance of 2-1/4Cr-1Mo steel after the initial heat treatment (austenitized at 927°C, cooled 50°C/hour to 704°C, held two hours, cooled 50°C/hour to 500°C, furnace cooled to room temperature).

Figure 8. Microstructural appearance of 2-1/4Cr-1Mo steel after the weld simulating heat treatment (austenitized at 1300°C for 10 minutes, oil quenched); a) Untempered and b) Tempered 10 hours at 760°C.

micrographs of the simulated HAZ microstructure and the simulated microstructure after the post-weld heat treatment, respectively.

Coupons subjected to GTA spot welding were prepared from material given the isothermal transformation heat treatment. Half of these coupons were then given a post-weld heat treatment of ten hours at 760°C . These two sets of coupons, in either the as-welded (AW) condition or the post-weld heat treated (PWHT) condition, were used as standard coupons for comparison with the simulated HAZ microstructure coupons. Figure 9 presents the geometry of the GTA-welded coupons. Coupons of the simulated microstructure were of similar geometry.

All testing was conducted in a glove box in which the atmosphere was purified by equipment specifically designed for that purpose. Oxygen and nitrogen levels in the argon atmosphere were controlled and kept below 200 ppm and 600 ppm, respectively, during testing. Crucibles made of 2-1/4Cr-1Mo steel were used for containment of the liquid lead-lithium alloys, as a means of avoiding dissimilar metal effects. The crucibles were heated by electric resistance elements and maintained to less than 1% deviation of

Figure 9. Schematic of GTA weld coupon geometry. Specimens for simulated HAZ microstructure were of the same dimensions.

temperature by using type K thermocouples. (See schematics of test apparatus used for placement of thermocouples). Actual liquid temperatures were monitored and recorded at the start of each test and at times of coupon or disk removal.

Once removed, corrosion specimens were sectioned, mounted, polished, and etched in 2% nital (=30 seconds) for optical examination. A microscope equipped with a video camera and its closed circuit television screen, calibrated for magnification, were used to measure depth of intergranular penetration. Penetration depths were systematically measured at each point of attack along the specimen surface, and a simple mean was calculated from the measurements.

Nitrogen levels in the liquid metals were measured, when feasible, during all testing. The level of nitrogen was determined by the micro-Kjedahl technique (22) and values ranged between 50 and 150 ppm. The lower limit of detectability, by this technique, given the 0.4cc sample size, is $50 \text{ ppm} \pm 50$. Figure 10 is a schematic of the sampling apparatus used for nitrogen analysis. The apparatus is comprised of a 304 stainless steel tube connected to a

Figure 10. Schematic of the lithium sampling technique and equipment.

chamber having a valve at either end. The chamber is evacuated before immersing the stainless steel tube into the liquid. Once immeresed, the lower valve is opened, drawing liquid into the tube. The tube is removed from the liquid, allowed to cool, and sectioned. The usable sample sections are placed in a air tight container and removed from the glove box for immediate nitrogen analysis.

2.2 Static Liquid Experiments

The procedure used in preparing coupons for static testing were as:

- a) slabs of material ($\approx 85\text{mm}$ by 150mm by 25mm) were isothermally transformed in a vacuum furnace,
- b) smaller slabs ($\approx 85\text{mm}$ by 5mm by 25mm) were sectioned from the larger pieces using an abrasion saw,
- c) the small slabs were austenitized at 1300°C and oil quenched,
- d) the slabs were rough ground to remove the surface layer affected by the heat treatment,
- e) a portion of each heat treated slab was given the post-weld heat treatment (10 hours at 760°C),
- f) coupons ($\approx 13\text{mm}$ by 13mm by 5mm) were sectioned from the small slabs,
- g) wire holes were drilled in the coupons, and coupon surfaces were fine ground on 600 grit silicon carbide paper.

The extent of the experimentation for the static testing is shown in Figures 11a and b, where test temperature and

Figure 11a. The temperature and lead-lithium compositions (up to 53w/o lead) investigated in the static liquid corrosion tests.

Figure 11b. The temperature and lead-lithium compositions (>53w/o lead) investigated in the static liquid corrosion tests.

composition are shown on the lead-lithium phase diagram. The apparatus used during the static testing is schematically drawn in Figure 12. Corrosion coupons were suspended by iron wire into a crucible containing one of the lead-lithium liquids. Periodically, coupons were removed and examined metallographically for intergranular penetration.

Figure 12. Schematic of the test apparatus used during the static liquid corrosion tests.

2.3 Dynamic Liquid Experiments (Spinning Disk)

Table III shows the extent of testing done utilizing the spinning disk apparatus.

TABLE III
Spinning Disk Test Matrix

SAMPLE HEAT TREATMENT	ANGULAR VELOCITY	
	2.23 ω (134RPM)	4.46 ω (268RPM)
austenitize at 1300°C 10 minutes, Oil Quench	X	X
Austenitize at 1300°C 10 minutes, Oil Quench, Temper 10 hours at 760°C	X	X

Note: Tests were conducted in liquid 17.6 w/o lead-lithium at 500°C for 400 hours.

The procedures used in preparing disks for the dynamic tests were similar to that used for the static tests; as before:

- a) blocks of material ($\approx 35\text{mm}$ by 35mm by 110mm) were isothermally transformed in a vacuum furnace,
- b) the blocks were machined into rounds ($\approx 30\text{mm}$ diameter and 150 mm long) and sectioned into disks ($\approx 30\text{mm}$

diameter and 5mm wide),

c) the disks were given the heat treatment to simulate a HAZ of a weldment (austenitize at 1300°C , oil quench),

d) the disks were machined into final disk dimensions (See Figure 13),

e) a portion of the disks were given the post-weld heat treatment (10 hours at 760°C),

f) the disks were finely ground to 600 grit (top and bottom) before exposure.

Figure 14 is a schematic of the apparatus used for testing. Velocity of the disk was monitored by using a light sensing cell which transmitted a signal to an oscilloscope for measurement and velocity determination. The signal was produced when a high intensity light was exposed to the cell. The exposure of the light to the cell was accomplished by placing a disk, with a slot cut in it, on the motor shaft, that turned the sample disk, and situating the cell and light in such a manner that, as the shaft turned, the disk's slot would expose the light to the cell once per revolution. Figure 15 is a schematic of the electronics used to provide and transmit the signal. Also shown are the output signals associated with the various parts in the circuit. The

Figure 13. Schematic of the dynamic test samples.

Figure 14. Schematic of the test apparatus used during the dynamic corrosion tests.

velocity of the disks varied less than 1% from the set velocity. The motor used to spin the disks was of a constant RPM type, and therefore gears were employed to obtain the desired speeds.

After 400 hours of exposure the disks were removed from the apparatus, cleaned in water and alcohol, sectioned, mounted, polished, and etched in 2% nital for optical analysis.

RESULTS and DISCUSSION

3.1 Conceptual Model

Previous data (24) have shown that the lithium penetration of 2-1/4Cr-1Mo steel is adequately described as parabolic in time; and consequently, important information regarding the corrosion mechanism(s) can be obtained from penetration data by an analysis of the temperature dependence. For parabolic time dependence:

$$P^2 = kt \quad (1)$$

where P is the intergranular penetration, t is the time, and k is the proportionality constant, dependent only on temperature:

$$k = k_0 \exp(-Q/RT) \quad (2)$$

If P is zero when t is zero, an expression equivalent to Equation (1) is:

$$P = (kt)^{1/2} \quad (3)$$

and the apparent activation energy for the corrosion process can be calculated from the slope of P versus the square root of time plots obtained from the various temperatures.

Specifically, when the natural logarithm of k , the proportionality constant, is plotted versus the reciprocal of absolute temperature, the slope of the resulting line is $-Q/R$, where Q is the activation energy and R is the gas constant.

When one mechanism is followed by a second this analysis is only correct for the initial mechanism; the time at which the subsequent mechanism initiates does not occur at zero, nor is the penetration zero at the time of initiation, and these facts must be accounted for in the analysis. This point is best clarified by deriving the parabolic penetration law from first principles.

Corrosion phenomena which obey a parabolic time dependence occur when the corrosion rate is controlled by steady-state diffusion. The diffusing species moves through a constant chemical potential gradient:

$$J = -D \frac{\Delta \mu}{\Delta x} \quad (4)$$

where J is the flux, D is the diffusivity, and $\Delta\mu/\Delta x$ is the chemical potential gradient. In the lithium corrosion of 2-1/4Cr-1Mo steel, the diffusing species is thought to be carbon, moving through the liquid lithium within the prior austenite grain boundaries of the steel. In this case, $\Delta\mu$ would be the chemical potential of carbon in the steel's carbide phase, minus the chemical potential of the carbon in the liquid lithium at the steel surface. The distance Δx would be the lithium penetration distance into the steel, P .

The penetration rate is simply related to the flux of the diffusing species:

$$\frac{dP}{dt} = -J\Omega \quad (5)$$

where Ω is the atomic volume of the diffusing species. Substituting Equation (5) into Equation (4), and replacing with P yields:

$$\frac{dP}{dt} = \Omega D \frac{\Delta\mu}{P} \quad (6)$$

Equation (6) is separable and integrable:

$$\int_{P_0}^P dP \approx \int_{t_0}^t (\Omega D \Delta \mu) dt \quad (7)$$

This expression, when integrated, becomes:

$$\begin{aligned} (P^2 - P_0^2) &= (2\Omega D \Delta \mu)(t - t_0) \\ &= k(t - t_0) \end{aligned} \quad (8)$$

Clearly, Equation (8) does not reduce to Equation (3) when P is not zero and t is not zero. This situation would occur if there was a break in the rate, either increasing or decreasing, after some initial rate; or if there were a delay time before corrosion began. A change in the penetration rate at intermediate times does occur for 2-1/4Cr-1Mo steel exposed to liquid 17.6w/o lead-lithium at temperatures from 440°C to 530°C.

The exact relation for such data is:

$$\int_0^{P_0} P dP + \int_{P_0}^P P dP \approx \int_0^{t_0} C_1 dt + \int_{t_0}^t C_2 dt \quad (9)$$

where $2C_1$ equals the initial rate constant k_1 and $2C_2$ equals

the secondary rate constant k_2 . Integrating and rearranging Equation (9), one obtains:

$$P^2 = 2C_2t + 2t_0(C_1 - C_2) \quad (10)$$

or

$$P^2 = k_2t + t_0(k_1 - k_2) \quad (11)$$

Since the term $t_0(k_1 - k_2)$ is a constant, an accurate rate constant for the secondary mechanism will be the slope obtained from a plot of penetration squared versus time.

Figure 16 is an Arrhenius plot of Anderson's (24) penetration data for both the initial and secondary mechanisms. The results derived from plots of penetration versus the square root of time are compared to those from plots of penetration squared versus time. The comparison reveals that analysis according to Equation (11), (P^2 vs t), yields a significantly lower activation energy for the secondary mechanism than does analysis according to Equation (3), (P vs $t^{1/2}$). The activation energy obtained for the initial mechanism was not significantly changed by re-analysis.

Figure 16. Arrhenius analyses of Anderson's data(24) utilizing Equation (3) (dashed lines) and Equation (10) (solid lines).

Reduction of the apparent activation energy for the secondary mechanism by a more correct data analysis requires a re-evaluation of the proposed mechanism for the lithium penetration. As shown in Figure 16, a more correct analysis of data allows the same mechanistic interpretation for both the initial and the secondary penetrations. Thus, the current interpretation of the intergranular penetration of 2-1/4Cr-1Mo steel by liquid lithium is that both the initial and secondary rates are controlled by the diffusion of carbon through liquid lithium. The change in penetration rate at intermediate times and temperatures ($\approx 440^{\circ}\text{C}$ to 530°C) represents only a decrease in the chemical potential gradient when epsilon carbide is converted to the more stable cementite.

3.2 Results of 500°C Static Coupon Experiments

In categorizing the effect lead may have on the corrosion phenomena observed in 2-1/4Cr-1Mo steel exposed to lead-lithium alloys, a matrix was devised (see Figure 12 and 13) to obtain needed information. Initially, the matrix consisted of experiments conducted at 500°C for four lead concentrations in lithium; 0, 17.6, 53, and 99.3 w/o lead. These experiments were for the purpose of gathering a broad base of information with which to compare and establish additional matrix points. All tests used coupons heat treated to simulate the HAZ of a weld, in both the as-welded and the post-weld heat treated conditions. In addition, these initial tests utilized standard GTA spot welds (as-welded and post weld heat treated) for comparison.

Figures 17, 18, and 19 are penetration results obtained from the first three lead concentration experiments. From these figures, one can see that the penetration of untempered coupons was similar for all three lead contents, and that the penetration versus time curves all have a parabolic nature. The curves also show untempered coupons being rapidly penetrated up to approximately 25 hours, after

Figure 17. The intergranular penetration of 2-1/4Cr-1Mo steel by liquid lithium at 500°C. The square symbols denote actual GTA weldments. The circles denote coupons given the heat treatment that simulates the HAZ microstructure of a weld.

Figure 18. The intergranular penetration of 2-1/4Cr-1Mo steel by liquid 17.6w/o lead-lithium at 500°C. The circles denote coupons given the heat treatment that simulates the HAZ microstructure of a weld.

Figure 19. The intergranular penetration of 2-1/4Cr-1Mo steel by liquid 53w/o lead-lithium at 500°C. The circles denote coupons given the heat treatment that simulates the HAZ microstructure of a weld.

which a slower penetration rate prevails. It should be noted that the post-weld heat treatment (10 hours at 760°C) was very effective in preventing intergranular penetration by the liquid lead-lithium alloys (up to 53 w/o lead) for times up to 1000 hours.

Figure 20 is a composite of the penetration versus time curves shown in Figures 17 through 19. It can be seen that all three curves could easily be enclosed in a relatively small scatter band. This band would be smaller than the relative standard deviations obtained for each test point. (See Appendix A). This suggests, at least for exposure times up to 1000 hours, at 500°C, and for lead concentrations up to 53 w/o lead, that the rate of attack by these liquids on 2-1/4Cr-1Mo steel is not significantly dependent upon the amount of lead in the lithium. It should be noted in stating this that 53 w/o lead in lithium corresponds to approximately 3.6 atomic percent (a/o) lead.

In all cases, the attack by the three liquid lead-lithium alloys on corrosion susceptible coupons occurred at prior austenite grain boundaries. This mode of attack is the same type as that reported in earlier work (24). Figure 21a and b are micrographs showing the effect

Figure 20. Composite plot of Figures 17 thru 19 comparing penetration results as a function of lead content. Lead content in the lithium, less than 53w/o, does not affect the rate of penetration at 500°C.

Figure 21. Intergranular penetration of simulated weldments of 2-1/4Cr-1Mo steel; a) SEM fractograph of a coupon exposed to liquid lithium at 400°C for 198 hours and b) Optical micrograph of coupon exposed to liquid lithium at 500°C for 1000 hours.

of lithium attack on susceptible coupons of 2-1/4Cr-1Mo steel. Figure 21a is a SEM micrograph showing the fracture surface of a corroded coupon. The coupon was exposed to liquid lithium at 400°C for 198 hours and then fractured at room temperature. Note that the fracture occurred along prior austenitic grain boundaries, giving the micrograph a "rock candy" appearance. Also seen in the micrograph are points of corrosion within the boundaries (see white arrows). Figure 21b is an optical micrograph on a plane perpendicular to the exposed surface, showing the typical prior austenite grain boundary attack by these liquid alloys.

Figure 22, 23, and 24 compare untempered coupons with post weld heat treated coupons after exposure to each of the three lead-lithium liquids (0, 17.6, and 53 w/oPb). Through such an optical comparison, it was concluded there were not gross differences in the corrosion behavior of the three lead-lithium alloys at 500°C. These figures show the effectiveness of post weld heat treatment (10 hours at 760°C) in preventing penetration of 2-1/4Cr-1Mo steel by lead-lithium liquids. Clearly, no penetration by the liquids occurred within 1000 hours when post weld heat treated coupons were exposed to liquid lead-lithium alloys

Figure 22. Optical micrographs of simulated weldments of 2-1/4Cr-1Mo steel exposed to liquid lithium at 500°C for 1000 hours; a) Untempered and b) Tempered 10 hours at 760°C.

Figure 23. Optical micrographs of simulated weldments of 2-1/4Cr-1Mo steel exposed to liquid 17.6w/o lead-lithium at 500°C for 1000 hours; a) Untempered and b) Tempered 10 hours at 760°C.

Figure 24. Optical micrographs of simulated weldments of 2-1/4Cr-1Mo steel exposed to liquid 53w/o lead-lithium at 500°C for 1000 hours; a) Untempered and b) Tempered 10 hours at 760°C.

at 500 C. It should also be noted that Figures 22 through 24 reveal no microstructural changes occurring in the coupons because of the presence of lead in the lithium.

The liquid 99.3 w/o lead-lithium alloy experiment showed no observable penetration in untempered or post weld heat treated coupons exposed for times up to 1000 hours. Also, no evidence of penetration was observed in GTA spot welded (as-welded and post weld heat treated) coupons exposed for 25 hours. Figure 25a and b are micrographs of the simulated HAZ microstructure, in both the untempered and post weld heat treated conditions, exposed to liquid 99.3 w/o lead-lithium alloy at 500°C for 1000 hours.

Figure 25. Optical micrographs showing lack of intergranular penetration in simulated weldments of 2-1/4Cr-1Mo steel exposed to liquid 99.3w/o lead-lithium at 500°C for 1000 hours; a) Untempered and b) Tempered 10 hours at 760°C.

3.3 Comparison of GTA Weld and Simulated HAZ Microstructures

Figures 17 and 18 not only show the penetration data for the corrosion susceptible simulated HAZ microstructures, but also the maximum penetration observed in as-welded GTA spot welds (see the square symbols in Figures 17 and 18). This maximum penetration was measured in that portion of the heat affected zone located next to the fusion zone. In addition to this observed maximum penetration, in the HAZ, less severe attack was seen in the fusion zone. Figure 26a is a micrograph of a corroded GTA spot weld coupon showing the severe attack in the HAZ. Figure 26b shows the lesser attack observed in the fusion zone.

In comparing simulated to actual GTA weld microstructures, it was found that corrosion appearance was similar, and that in all cases GTA weld penetration was within the standard deviations calculated for penetration of simulated weld microstructures exposed to similar liquids and for comparable times.

Post weld heat treated GTA welds exhibited lead-lithium corrosion behavior similar to that seen in the simulated

Figure 26. Intergranular penetration of a GTA weldment exposed to liquid lithium at 500°C for 1000 hours; a) HAZ and b) Fusion zone.

HAZ microstructures. A simple post-weld heat treatment effectively prevented penetration. Figure 27a and b are micrographs of a post weld heat treated GTA welds after exposure to lithium at 500°C for more than 1000 hours. Figure 27a is of the HAZ right next to the fusion zone, while Figure 27b is of the fusion zone. Note the lack of penetration of these post-weld heat treated samples.

It should be reiterated that the region of maximum penetration in a weld is the region that the unorthodox heat treatment (1300°C , oil quench) tries to approximate microstructurally; and because the heat treatment only simulates a small region of a zone that has a large thermal gradient associated with it, differences can arise in the observed behavior between actual GTA welds and coupons given the special heat treatment. An example of just such a difference is the reported (24) attack at lath boundaries in post weld heat treated GTA welds and the lack of attack observed in this investigation's post weld heat treated simulated HAZ microstructures. The difference can be explained with the aid of Figure 28, a schematic diagram showing the region simulated by the special heat treatment (maximum penetration region or Region A) and the region of interlath attack observed by Anderson (24) in post weld heat

Figure 27. Lack of intergranular penetration observed in post-weld heat treated (10 hours at 760°C) GTA weldment exposed to liquid lithium at 500°C for 1000 hours; a) HAZ and b) Fusion zone.

Figure 28. Schematic of the proposed reason for the lack of intergranular penetration observed in post-weld heat treated (10 hours at 760°C) simulated weldment coupons.

treated GTA welds (Region B). It can easily be rationalized that if interlath attack was not observed in Region A of the actual post weld heat treated GTA welds then the same should be true of coupons given a post weld heat treatment after the special heat treatment.

Anderson explained that interlath penetration was due to the presences of Mo_2C in the microstructure. He supported his theory by stating that the HAZ of a weldment could be thought of as a quenched microstructure. With this assumption he then utilized the Baker and Nutting carbide stability diagram (Figure 5) and the thermodynamic stability diagram of certain carbides in carbon saturated lithium (Figure 6) to show that Mo_2C was still present after the 10 hour 760°C post-weld heat treatment, and that it was less stable with respect to Li_2C_2 at 500°C .

3.4 Comparison of Static Liquid Test Data with Previous Data

Figure 29 compares Anderson's (24) data (open circles) with the data obtained in this investigation (the data of Figure 18 shown here as a solid line) for as-quenched 2-1/4Cr-1Mo steel exposed to liquid 17.6 w/o lead-lithium at 500°C. It can be seen that the initial penetration rates obtained in these two studies are remarkably similar. However, penetration at times greater than about 25 hours were significantly slower in the previous study than in this investigation. A possible cause for the observed deviation could be the means by which the coupons were initially heated treated to simulate the corrosion susceptible microstructure. Anderson used an induction furnace for heating, and time to temperature before quenching was approximately one minute. This investigation's coupons were heated in a glo-bar furnace where the time at temperature before quenching was about ten minutes.

Dissolution kinetics for carbides should not be affected by the method in which the material is heated; however the time, at temperature should have a marked affect. Because the time to temperature is so short with

Figure 29. Comparison of penetration data for simulated weldments of 2-1/4Cr-1Mo steel coupons in liquid 17.6w/o lead-lithium at 500°C.

the induction furnace, the carbides present (Mo_2C , M_3C) after the isothermal heat treatment might not dissolve completely, and elements from the dissolving carbides (Cr , Mo , Fe) may not have sufficient time for diffusing a great distance away from the carbide sites. In addition, because of the heat input the remaining carbides could begin to transform to more stable compositions (M_{23}C_6 , Cr_7C_3 , and M_6C) following the sequence presented by Baker and Nutting (31). Conceivably, the partially dissolved and transformed carbide could rapidly transform to a stable morphology when in the presence of the liquid alloy at 500°C . Therefore, rapid transformation would be due to the close proximity of the stable carbide forming elements (Cr , Mo) and due to the fact that transformation to a more stable form began during the up quench.

3.5 Temperature Dependence of the Penetration of 2-1/4Cr-1Mo Steel by Lead-Lithium Liquids

Although initial studies in lithium, 17.6 w/o, and 53 w/o lead-lithium liquids suggested that the rates and mechanisms involved in the corrosion of 2-1/4Cr-1Mo steel might be similar, further verification was desired. Thermal analysis was deemed necessary, and the test matrix was expanded to include penetration experiments at various temperatures in liquid lithium and liquid 53 w/o lead-lithium. The liquid 17.6 w/o lead-lithium was not examined because thermal analysis had been done by Anderson (24) in his investigation of this alloy.

Figures 30 through 33 are the penetration versus time curves obtained for the tests conducted in the two liquids. Taking the rate constant, $k(\mu\text{m}^2/\text{s})$ for the data in Figures 30 through 33 (see Equation 11) and plotting it versus the reciprocal of the absolute temperature produces an Arrhenius plot, seen in Figure 34. Previous results for the 17.6 w/o lead-lithium liquid are plotted with the recent data for both lithium and 53 w/o lead-lithium liquids so that an easy comparison can be made.

Figure 30. Penetration data of simulated weldments (oil quenched from 1300°C) as a function of time and temperature in liquid lithium.

Figure 31. Penetration data of simulated weldments (oil quenched from 1300°C) as a function of time and temperature in liquid lithium.

Figure 32. Penetration data of simulated weldments (oil quenched from 1300°C) as a function of time and temperature in liquid 53w/o lead-lithium.

Figure 33. Penetration data of simulated weldments (oil quenched from 1300°C) as a function of time and temperature in liquid 53w/o lead-lithium.

Figure 34. Comparison of apparent activation energies as a function of lead concentration in liquid lithium.

Activation energies for the initial and secondary rates by liquid lithium were found to be 32 kJ/mole and 30 kJ/mole, respectively. Corresponding values for the 53 w/o lead-lithium liquid were found to be 24 kJ/mole and 69 kJ/mole.

The activation energies for the penetration by liquid lithium and 53 w/o lead-lithium liquid were not significantly different from those calculated for penetration by a 17.6 w/o lead-lithium liquid. The conclusion can therefore be reached that the presence of up to 53 w/o (≈ 3.6 at/o) lead in the liquid lithium does not markedly affect the mechanisms by which the liquid lithium attacks the steel. As was stated before, the present theory on the mechanism associated with initial rapid penetration rate and the slower secondary penetration rate is that both rates are controlled by the diffusion of carbon through the liquid filled corroded austenite grain boundaries. The difference between the two rates corresponds to a decrease in the chemical potential gradient when epsilon carbide is converted to the more stable cementite.

The rate at which the various lead-lithium liquids corroded 2-1/4Cr-1Mo steel can also be compared using

Figure 34. From these data, it is apparent that the initial and secondary rates of attack were found to decrease when lead is added to the lithium liquid. However, the decrease is not systematic with respect to the concentration of the lead. These data suggest that lead must inhibit the diffusion of the carbon through the corroded grain boundaries.

3.6 The Anomalous Behavior of 500°C

Table IV is a comparison of the penetration rate constants for three lead-lithium liquids at three temperatures (480, 500, and 530°C) and for both mechanisms. From the table, one notes that the 500°C rate constants are approximately one order of magnitude less than those calculated for the other two temperatures.

TABLE IV
Comparison of Rate Constants

Temperature (°C)	w/o Lead	Rate Constant ($\mu\text{m}^2/\text{s}$)	
		initial	secondary
480	0	13.8	0.758
	17.6	3.14	0.139
	53	5.42	0.3.72
500	0	0.556	0.0186
	17.6	0.867	0.0217
	53	0.227	0.0251
530	0	26.6	0.847
	17.6	7.92	0.217
	53	7.94	0.653

Note: rate constant values for 17.6w/o lead at 480 and 530°C were taken from Anderson's work(24).

By plotting the rate constants calculated from exposure of 2-1/4Cr-1Mo steel to lead-lithium liquids at 500°C on an Arrhenius plot (as was previously done for other temperatures in Figure 34) one can graphically see that the data for 500°C fall well below the values established by the other temperatures (see Figure 35). This observed anomaly is not an isolated case; Anderson (24) also observed the anomaly but believed it to be caused by slight microstructural differences in the steel samples.

Great care was taken in the present work to assure uniformity of specimen microstructure, and it is believed that anomalous behavior at 500°C is a real phenomenon, controlled by competitive processes yet to be determined.

A proposed model which consistently explains the phenomenon at 500°C has been constructed. A schematic representation of the time and temperature dependence of this phenomenon is presented in Figure 36, and a schematic drawing of grain boundary carbide attack in accordance with this model is presented in Figure 37. If it is assumed that a protective corrosion product causes the penetration of 2-1/4Cr-1Mo steel by lead-lithium liquids to be a minimum at

Figure 35. Arrhenius plot showing anomalously low rate constant values for corrosion data at 500°C.

Figure 36. A possible explanation for the anomalously low penetration rate of 2-1/4Cr-1Mo steel by lead-lithium liquids at 500°C. A protective corrosion product dissolves rapidly in lithium at temperatures greater than 500°C, and forms slowly at temperatures less than 500°C.

LIQUID-METAL INTERFACE

Figure 37. A hypothetical model for the anomalously low penetration rate of 2-1/4Cr-1Mo steel by lead-lithium liquids at 500°C . A protective corrosion product dissolves rapidly at temperatures greater than 500°C , and forms slowly at temperatures less than 500°C .

500°C, then the accumulation rate of that product can be assumed to be dependent on temperature as indicated in Figure 37. The corrosion product accumulation rate decreases at temperatures greater than 500°C because the solubility of that product in the liquid metal or alloy increases rapidly with increasing temperature, dissolving away the product as it forms. On the other hand, the corrosion product accumulation rate decreases at temperatures lower than 500°C because the formation rate is a typical thermally activated chemical reaction, and the reaction rate rapidly decreases with decreasing temperature.

3.7 The Initial Penetration Rate's Relationship with Epsilon Carbide

A brief study was undertaken during this investigation to substantiate the proposed dependence of the initial rapid penetration rate on the presence of epsilon carbide in the microstructure. Simulated HAZ microstructures of 2-1/4Cr-1Mo steel were given a temper of 25 hours at 480°C before exposure to liquid lithium at 480°C. This temper was intended to remove, by transformation, any epsilon carbide present in the microstructure while leaving the cementite. If the initial rapid penetration were indeed caused by attack of epsilon carbide, then such a specimen should be penetrated from the beginning at a rate comparable to what has been termed the "secondary mechanism" rate.

Figure 38 shows the results from the above experiment. Also plotted are the penetration data for untempered exposed to lithium at 480°C (see Figure 30). Figure 38 clearly shows the absence of the initial rapid penetration in the tempered coupons. These data lend significant credibility to the hypothesis by Anderson (24) that the initial rapid penetration was associated with the presence of epsilon carbide in the microstructure.

Figure 38. Penetration data comparison of simulated weldment coupons of 2-1/4Cr-1Mo steel exposed to liquid lithium at 480°C.

3.8 Dynamic Corrosion Testing

The spinning disk tests performed during the dynamic corrosion testing were for the purpose of determining whether the corrosion mechanism observed in the static tests was sensitive to velocity of the environment. Understanding of the basic theory of the spinning disk is important and therefore will be given before presenting the data.

Simply explained, the theory states that the controlling process is the diffusion of a specie through a laminar boundary layer that exists between the disk surface and the bulk liquid. In addition, the theory dictates that the corrosion mechanism be liquid diffusion. From this simplified explanation it can be seen that a chemical potential gradient has to exist across the boundary layer for diffusion into the bulk liquid to occur; Ficks' first law would apply and the flux of atoms across the boundary layer would be dependent upon the boundary layer thickness.

However, velocity of the disk is the primary parameter of interest and not the boundary layer thickness. Therefore, a function relating the two is necessary.

Previous investigators (42-43), using a mathematical solution, have shown that the boundary layer thickness is inversely proportional to the square root of the disk's angular velocity. Hence they obtained an expression relating the flux of atoms diffusing across the boundary layer to the angular velocity of the disk.

Because the penetration is proportional to the flux of atoms diffusing away from the corrosion front, it must also be dependent upon the boundary layer thickness and hence proportional to the square root of the angular velocity of the disk. By plotting the penetration in a given time against the angular velocity of the disk, the sensitivity of the corrosion process can be seen. Martins (44) explained that in this analysis an increasing or decreasing penetration with increasing angular velocity would indicate a sensitivity of the corrosion process to a dynamic environment. Simply put, the corrosion process would be dependent upon the diffusion of a given specie across the boundary layer. Whereas, an insensitivity or no dependence of the corrosion process on the boundary layer thickness would be shown by neither an increase or decrease in penetration with an increase in angular velocity of the disk.

Figure 39 presents the results from the preliminary work performed on the effect of velocity on the corrosion of 2-1/4Cr-1Mo steel by a 17.6 w/o lead-lithium liquid. The figure shows that the penetration of simulated HAZ microstructures was quite insensitive to the angular velocity, at least in the narrow range of velocities tested. Figure 39 also emphasizes that post weld heat treatment of 2-1/4Cr-1Mo steel was very effective in preventing penetration by the liquid. Previous work (42,43) on dynamic corrosion similar to that done here suggests that the penetration should be independent of velocity when the observed attack is of intergranular nature.

Figures 40a and b are SEM micrographs of spinning disk specimen surfaces after exposure for 400 hours to the dynamic liquid. Figure 40a is of the post weld heat treated specimen and Figure 40b is of the untempered specimen. Micrograph 40a shows a surface with no apparent signs of attack. Note that the 600 grit polishing scratches are still visible on the disk surface. Micrograph 40b shows a severely attacked surface that appeared etched when examined by the naked eye.

Figure 39. The effect of post-weld heat treatment and velocity on the intergranular penetration of simulated weldment 2-1/4Cr-1Mo steel by a 17.6w/o lead-lithium liquid. Corrosion is seen to be greatly reduced by the post-weld heat treatment, and to be insensitive to low angular velocity.

Figure 40. SEM micrographs of disk surface after exposure for 400 hours at an angular velocity of 4.46 revolutions per second ; a) Untempered and b) Tempered 10 hours at 760°C.

Figures 41a and b are optical micrographs of the same two disks; however, the micrographs are taken perpendicular to the exposed surface. It can be seen that the observed corrosion in untempered disks was similar to that observed in the untempered static corrosion coupons, and that the post weld heat treatment effectively prevented intergranular penetration.

From the experiment performed, 2-1/4Cr-1Mo steel was found to be insensitive to velocity in the range tested. However, one must remember that the test excluded thermal gradients which are part of the design criteria during the normal operation of a inertia confinement fusion reactor.

Figure 41. Intergranular penetration and lack of penetration observed in simulated weldments of 2-1/4Cr-1Mo steel disks spun at 4.46 revolutions per second for 400 hours and in a 17.6w/o lead-lithium liquid at 500°C; a) Untempered and b) Tempered 10 hours at 760°C.

CONCLUSIONS

1) The corrosion mechanism, the dissolution of epsilon carbide and cementite and subsequent diffusion of the carbon through the corroded grain boundary, by which 2-1/4Cr-1Mo steel is attacked by lead-lithium liquids does not vary for compositions up to 53 w/o lead. In addition, initial and secondary attack only differs in the rate at which the attack proceeds. The difference is believed to be associated with the stability of the carbides present. The initial rate is dependent on the dissolution of epsilon carbide, while the secondary rate is dependent on the dissolution of cementite.

2) Previous Arrhenius analysis of lithium corrosion data yielded an erroneously high activation energy for long exposure times. A corrected analysis yields an activation energy like that obtained for short exposure times, and the penetration of 2-1/4Cr-1Mo steel by a 17.6 w/o lead-lithium liquid can now be explained as controlled by the diffusion of carbon in liquid lithium over all exposure times.

3) The untempered bainitic microstructure of 2-1/4Cr-1Mo steel is highly susceptible to intergranular penetration by lead-lithium liquids containing up to 53 w/o lead.

4) A tempering heat treatment at 760°C for 10 hours is very effective in preventing the intergranular attack of as-quenched 2-1/4Cr-1Mo steel by these lead-lithium liquids for times up to 1000 hours at 500°C.

5) There were no gross differences in appearance or mode of attack among untempered specimens exposed to the various lead-lithium liquids at 500°C; nor were there any differences observed in tempered coupons exposed to these same liquids.

6) Neither as-quenched nor tempered 2-1/4Cr-1Mo steel is intergranularly attacked within 1000 hours by a 99.3 w/o lead-lithium liquid at 500°C.

7) The intergranular penetration rates of 2-1/4Cr-1Mo steel are reduced by additions of lead at temperatures from 300°C to 600°C.

8) Anomalous low corrosion of 2-1/4Cr-1Mo steel by lead-lithium liquids occurs at 500°C. This behavior may be related to the formation of a corrosion product.

9) The intergranular penetration of corrosion susceptible 2-1/4Cr-1Mo steel by 17.6 w/o lead-lithium liquid is not increased by angular velocities of the disk up to 4.46 revolutions per second (268 RPM).

APPENDIX A

Penetration Data for 2-1/4Cr-1Mo Steel
Exposed to Lead-Lithium Liquids
for Various Times and Temperatures

SAMPLE	TIME (hours)	MIN. P (mm)	MAX. P (mm)	MEAN (mm)	S.D. (mm)
LITHIUM-573 K					
1-11	1	0.064	0.250	0.156	0.093
1-12	4	0.093	0.448	0.245	0.142
1-13	9	0.048	0.720	0.393	0.175
1-14	25	0.150	0.816	0.457	0.145
LITHIUM-623 K					
2-1	1	0.035	0.461	0.215	0.136
2-2	2	0.026	0.506	0.275	0.140
2-3	4	0.070	0.774	0.355	0.163
2-4	9	0.160	1.184	0.561	0.280
2-5	16		CORRODED THRU		
LITHIUM-673 K					
1-1	3	0.040	0.712	0.274	0.206
1-2	9	0.056	0.816	0.463	0.230
1-3	25		CORRODED THRU		
1-4	49	0.800	1.312	0.974	0.232
LITHIUM-753 K					
3-1	1	0.016	1.312	0.261	0.382
3-2	4	0.112	1.040	0.558	0.304
3-3	6	0.147	1.696	0.798	0.435
3-4	16	0.128	1.952	0.801	0.689
3-7	24	0.275	3.136	0.912	0.789
3-5	48	0.403	1.843	0.876	0.556
3-6	81	0.416	1.664	0.929	0.504
3-8	121		CORRODED THRU		

SAMPLE	TIME (hours)	MIN. P (mm)	MAX. P (mm)	MEAN (mm)	S.D. (mm)
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LITHIUM-753 K Tempered 25 hours at 753 K

Z-1	1	0.011	0.045	0.022	0.010
Z-2	9	0.016	0.152	0.044	0.040
Z-3					
Z-4					
Z-5	121	0.040	0.184	0.104	0.054
Z-6	225	0.040	0.190	0.123	0.045

LITHIUM-773 K

C-1	4	0.048	0.189	0.122	0.058
C-2	16	0.174	0.371	0.240	0.086
C-3	25	0.072	0.368	0.184	0.091
C-4	49	0.160	0.240	0.184	0.034
C-5	100	0.107	0.349	0.226	0.082
C-6	144	0.152	0.384	0.248	0.070
C-7	225	0.136	0.413	0.264	0.084
C-8	496	0.131	0.296	0.249	0.042
C-9	625	0.203	0.429	0.304	0.058
C-10	1024	0.206	0.512	0.335	0.074

NO CORROSION OBSERVED IN PWHT COUPONS

GTA COUPONS - LITHIUM-773

D-1	25	0.104
D-2	1024	0.395

NO CORROSION OBSERVED IN PWHT COUPONS

LITHIUM-803 K

6-1	1/4	0.026	0.280	0.125	0.066
4-11	1	0.048	0.646	0.313	0.327
6-2	4	0.154	0.454	0.332	0.110
6-4	25	0.147	0.568	0.334	0.136
6-3	49	0.160	1.112	0.505	0.351
6-5	81	0.075	1.584	0.576	0.499

SAMPLE	TIME (hours)	MIN. P (mm)	MAX. P (mm)	MEAN (mm)	S.D. (mm)
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LITHIUM-823 K

12-11	1	0.016	0.082	0.053	0.023
12-12	4	0.045	0.504	0.140	0.151
12-13	9	0.077	0.286	0.164	0.065
12-14	25	0.176	0.267	0.221	0.031
12-15	48	0.117	0.528	0.289	0.133

LITHIUM-873 K

11-6	1/4	0.014	0.240	0.060	0.075
11-7	1	0.053	0.154	0.105	0.031
11-8	4				
11-9	9	0.053	0.416	0.197	0.093
11-10	25	0.080	0.880	0.400	0.204

17.6w/o LEAD-LITHIUM-773

F-1	4	0.037	0.043	0.040	0.003
1	4.5	0.050	0.112	0.080	0.038
F-2	16	0.125	0.480	0.307	0.178
2	16	0.118	0.512	0.285	0.170
3	25	0.091	0.275	0.181	0.070
4	49	0.109	0.272	0.190	0.077
F-5	100	0.187	0.440	0.273	0.145
5	100.5	0.109	0.544	0.254	0.134
F-6	144				
F-7	225				
F-8	400	0.234	0.635	0.363	0.131
F-9	625	0.176	0.680	0.347	0.110
F-10	1024	0.296	0.496	0.358	0.072

NO CORROSION OBSERVED IN PWHT COUPONS

GTA COUPONS - 17.6w/o LEAD-LITHIUM

D-7	25	0.064
D-9	1024	0.243

NO CORROSION OBSERVED IN PWHT COUPONS

SAMPLE	TIME (hours)	MIN. P (mm)	MAX. P (mm)	MEAN (mm)	S.D. (mm)
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53w/o LEAD-LITHIUM-713 K

11-1	1/4	0.037	0.093	0.060	0.018
11-2	1	0.027	0.720	0.148	0.177
11-3	4	0.074	0.363	0.221	0.073
11-4	9	0.208	0.536	0.360	0.073
11-5	25	0.216	1.280	0.659	0.184

53w/o LEAD-LITHIUM-733 K

12-6	1/4	0.030	0.205	0.085	0.043
12-7	1	0.037	0.418	0.155	0.105
12-8	4	0.064	0.328	0.211	0.072
12-9	9	0.101	1.456	0.450	0.400
12-15	25	0.227	1.472	0.716	0.358

53w/o LEAD-LITHIUM-753 K

7-1	1/4	0.030	0.408	0.109	0.085
7-2	1	0.037	0.613	0.197	0.181
7-3	4	0.083	0.840	0.272	0.219
7-4	9	0.059	1.371	0.418	0.357
7-5	25	0.122	0.760	0.348	0.226
7-6	49	0.176	1.208	0.506	0.273
7-7	81	0.088	1.560	0.490	0.480
4-12	120.5	0.138	1.464	0.528	0.361

53w/o LEAD-LITHIUM-773 K

5-11	1	0.016	0.120	0.067	0.045
5-1	4	0.077	0.256	0.152	0.061
5-12	4	0.088	0.166	0.128	0.037
5-13	9	0.112	0.240	0.152	0.440
5-2	16				
5-3	25	0.048	0.240	0.127	0.056
5-14	25	0.091	0.157	0.117	0.028
5-4	49	0.126	0.608	0.225	0.119
5-5	100	0.072	0.304	0.177	0.070
5-6	144	0.072	0.280	0.155	0.062
5-7	256	0.088	0.528	0.248	0.089
5-8	400	0.120	0.536	0.312	0.115

SAMPLE	TIME (hours)	MIN. P (mm)	MAX. P (mm)	MEAN (mm)	S.D. (mm)
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53w/o LEAD-LITHIUM-773 K Continued

5-9	625	0.176	0.517	0.278	0.093
5-10	990	0.216	0.584	0.325	0.099

53w/o LEAD-LITHIUM-803 K

0-1	1/4	0.016	0.275	0.101	0.070
3-9	1	0.024	1.344	0.167	0.247
3-10	4	0.016	1.472	0.204	0.285
3-13	6	0.030	1.504	0.313	0.356
3-14	16	0.064	1.280	0.358	0.358
3-15	24	0.051	1.280	0.330	0.358
3-16	33	0.064	1.740	0.396	0.525
4-9	48	0.107	1.253	0.439	0.400
4-10	81	0.243	1.090	0.497	0.270

53W/O LEAD-LITHIUM-843 K

6-6	1/4	0.064	0.435	0.140	0.085
6-7	1	0.056	0.272	0.184	0.087
6-8	9	0.074	0.502	0.269	0.125
6-9	25	0.029	1.128	0.326	0.415
6-10	49	0.090	1.768	0.471	0.727

53w/o LEAD-LITHIUM-873 K

12-1	1/4	0.026	0.090	0.060	0.023
12-2	1	0.085	0.176	0.130	0.034
12-3	4				
12-4	16.5	0.200	0.472	0.297	0.072
12-5	24	0.160	0.752	0.403	0.144

99.3w/o LEAD-LITHIUM-773 K

NO PENETRATION OBSERVED IN EITHER CORROSION SUSCEPTIBLE OR
PWHT COUPONS FOR TIMES UP TO 1000 HOURS

APPENDIX B

Calculated Rate Constants

temperature (K)	rate constant ² ($\mu\text{m}^2/\text{s}$)
LITHIUM	
573 (I)	2.64
623 (I)	9.61
673 (I)	5.42
753 (I)	13.8
(S)	7.58×10^{-1}
773 (I)	5.56×10^{-1}
(S)	1.86×10^{-2}
803 (I)	26.6
(S)	8.47×10^{-1}
823 (S)	4.44×10^{-1}
873 (S)	1.72
17.6w/o LEAD-LITHIUM	
773 (I)	8.67×10^{-1}
(S)	2.17×10^{-2}

TEMPERATURE (K)	RATE CONSTANT ² ($\mu\text{m} / \text{s}$)
53w/o LEAD-LITHIUM	
713 (I)	4.69
733 (I)	5.69
753 (I)	5.42
(S)	3.72×10^{-1}
773 (I)	2.27×10^{-1}
(S)	2.51×10^{-2}
803 (I)	7.94
(S)	6.53×10^{-1}
843 (S)	1.08
873 (S)	1.76

note: (I) Represents Initial Rate
(S) Represents Secondary Rate

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