CONF-860421--15

CONF-860421--15

DE86 009152

Corrosion of Ferrous Alloys Exposed to Thermally
Convective Pb-17 at. % Li*

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MASTER

Abstract

A type 316 stainless steel thermal convection loop with type 316 stainless steel coupons and a Fe-9 Cr-1 Mo steel loop containing Fe-12 Cr-1 MoVW steel specimens circulated molten Pb-17 at. % Li at a maximum temperature of 500°C. Specimens were exposed for greater than 6000 h. Mass loss and surface characterization data were compared for these two alloys. At any particular exposure time, the corrosion of type 316 stainless steel by Pb-17 at. % Li was more severe, and of a different type than that of similarly exposed Fe-12 Cr-1 MoVW steel. The austenitic alloy suffered nonuniform penetration and dissolution by the lead-lithium, whereas the Fe-12 Cr-1 MoVW steel tended to be more uniformly corroded. The presence of a ferritic layer on the type 316 stainless steel, and its susceptibility to spalling during specimen cleaning, were shown to be important in evaluating the data and in comparing corrosion losses for the two types of alloys. A model for the nonuniform penetration of type 316 stainless steel by Pb-17 at. % Li was suggested.

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Corrosion of Ferrous Alloys Exposed to Thermally Convective Pb-17 at. % Li*

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Introduction

In recent years, molten Pb-17 at. % Li has received consideration as a possible tritium breeding fluid for fusion reactors [1]. This material provides effective tritium breeding and is safer, relative to molten lithium, under accident conditions because it is less reactive with air and water. However, the corrosion of typical containment alloys by Pb-17 at. % Li appears problematical. Corrosion studies performed to date [2-5] have shown that Pb-17 at. % Li is generally more aggressive than molten lithium, but additional work is required to completely characterize the corrosion processes in lead-lithium and to develop approaches for decreasing its corrosiveness by compositional modifications or by alloy development. To this end, the present paper contains results and analyses from relatively long-term exposures of both type 316 stainless steel and Fe-12 Cr-1 MoVW steel to thermally convective Pb-17 at. % Li at 500°C. These structural alloys represent the principal classes of steel being considered for fusion applications.

Experimental procedures

The experiments were conducted in thermal convection loops that allowed the corrosion specimens to be inserted and removed without stopping the

*Research sponsored by the Office of Fusion Energy, U.S. Department of Energy under contract DE-AC05-84OK21400 with Martin Marietta Energy Systems, Inc.

lead-lithium flow. Such loops have also been used for lithium studies and are described in detail elsewhere [6]. Coupons were positioned in both the hot and cold legs of the loops. Generally, a cluster of specimens was placed at the top of the hot leg in order to maximize the number of coupons being exposed at the highest loop temperature. The type 316 stainless steel specimens were exposed in a loop of matching base composition; however, the loop had circulated lithium for over 10,000 h prior to operation with lead-lithium. The Fe-12 Cr-1 MoVW steel specimens were contained in a loop made from Fe-9 Cr-1 Mo steel that had not seen previous service. Both loops circulated Pb-17 at. % Li at a maximum temperature of 500°C, but the temperature differential was higher for the ferritic steel loop (130 versus 80°C). The flow velocity was about 10 mm/s, which approximately simulates the semistagnant condition of the tritium breeding fluid. The Pb-17 at. % Li used in these loops was prepared by first purging pure molten lead with hydrogen, adding the appropriate amount of lithium in molten form, and then holding the mixture at 730°C for 3 to 4 h in a molybdenum-lined pot.

Residues of lead-lithium remaining on the specimens after exposure were removed by soaking in molten lithium (between 230 and 290°C) for about 6 h followed by water rinsing and drying. The specimens were then repeatedly weighed and reimmersed in lithium until their weights became constant. The temperature of the lithium was sufficiently low so that corrosion effects during specimen cleaning were negligible. However, as described below, the use of lithium to remove residual lead-lithium sometimes resulted in the partial or complete separation of the corrosion layer that formed on

type 316 stainless steel during loop exposure. Therefore, in most cases, once a specimen was removed from a loop and cleaned, it was not again exposed to the lead-lithium. The specimens grouped at the top of the hot leg at 500°C were selectively removed and replaced with fresh specimens at specific time intervals to measure corrosion losses as a function of exposure time.

The composition of the mill-annealed type 316 stainless steel used in this study was 65 Fe, 17 Cr, 12 Ni, 2 Mo, 2 Mn, 2 Si, 0.08 C (wt %). The Fe-12 Cr-1 MoVW steel consisted of 85 Fe, 12 Cr, 1 Mo, 0.5 Ni, 0.6 Mn, 0.4 W, 0.3 V, 0.2 Si, 0.2 C (wt %). These specimens were normalized and tempered (0.5 h at 1050°C, 2.5 h temper at 780°C, in flowing argon) prior to exposure to the molten lead-lithium.

Results

The corrosion of these ferrous alloys by thermally convective Pb-17 at. % Li was characterized and analyzed as a function of time using weight and thickness changes, observations of corroded surfaces, and measurements of environmentally-induced compositional changes. The microstructural and compositional data were obtained from optical and scanning electron microscopy and energy- and wavelength-dispersive x-ray analyses.

An important aspect of this investigation concerned the removal of residual lead-lithium from the exposed specimens and its effect on weight change and microstructural data. The typical condition of an exposed type 316 stainless steel surface that was mounted and polished without

cleaning is shown in Fig. 1, which includes backscattered electron images of the polished cross section and the corresponding x-ray elemental images. These electron microprobe data revealed that the stainless steel surface was severely infiltrated by lead (and presumably lithium) and that the resulting "corrosion layer" was somewhat depleted in nickel and chromium. In contrast, examination of an uncleaned coupon of Fe-12 Cr-1 MoVW steel that was similarly exposed to Pb-17 at. % Li revealed no evidence of any internal penetration or localized corrosion (see Fig. 2).

Micrographs of uncleaned specimens must be compared to those of specimens cleaned by the typical procedure to determine the effects of the lithium soak on existing surface morphology. A comparison of soaked and unsoaked type 316 stainless steel specimens is shown in Fig. 3. Both specimens were exposed at 500°C for the same period of time in the stainless steel lead-lithium loop. The micrographs indicated that the lithium cleaning of the exposed type 316 stainless steel removed a significant amount of material as evidenced by the voids in the corrosion layer shown in Fig. 3(b). A large fraction of the material removed by the lithium soak was residual lead; electron microprobe analysis of a polished cross section from an exposed and cleaned type 316 stainless steel specimen showed very little lead in the corrosion layer relative to the amount apparent in Fig. 1. However, the amount of steel constituents removed during cleaning with lithium is not known. Some of the corrosion layer is probably also lost during the cleaning process. In one case, most of the corrosion layer was removed from a type 316 stainless steel specimen after repeated exposures to thermally convective Pb-17 at. % Li and the standard (lithium and water) cleaning procedure [7].

The corrosive attack of type 316 stainless steel exposed to thermally convective Pb-17 at. % Li was preferential with respect to the main alloy constituents. A scanning electron micrograph of a typical specimen is shown in Fig. 4. Energy dispersive x-ray analyses of such surfaces and of cross sections showed that the remaining material in the corrosion layer was depleted in nickel and chromium relative to the starting composition of the steel (in agreement with the qualitative electron microprobe results of Fig. 1). This preferential depletion was confirmed by the quantitative results from an electron microprobe cross sectional analysis of the specimen exposed for 6384 h at 500°C; the wavelength dispersive x-ray data are given in Table 1. It is interesting to note that, although the compositional data for the specimen exposed for 6384 h revealed rather uniform (and low) concentrations of nickel and chromium throughout the corrosion layer, the measured concentrations of these elements in a corrosion layer formed after 2472 h of exposure varied between the values noted in Table 1 and those levels typically found in the starting material. In direct contrast to the case of type 316 stainless steel, no localized corrosion of the exposed Fe-12 Cr-1 MoVW steel specimens was observed during examination by optical and scanning electron microscopy. Polished cross sections of Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li for as long as 6048 h were similar to that shown in Fig. 2, regardless of whether a lithium rinsing procedure was used.

Although attack of the Fe-12 Cr-1 MoVW steel was quite uniform, significant amounts of material was dissolved during loop exposures; weight loss

measurements versus exposure time for this steel are shown in Fig. 5. The data represent measurements from individual coupons, which were cleaned in the standard manner after loop exposure and not reexposed. The mass change results for the type 316 stainless steel specimens exposed to Pb-17 at. % Li at 500°C in the stainless steel loop were obtained in a similar way and are also presented in Fig. 5. The weight change data for the two types of alloys revealed much greater weight losses for the type 316 stainless steel specimens, and also indicate different weight loss kinetics. However, as will be discussed below, there is an uncertainty in the type 316 stainless steel weight losses that results from the porous corrosion layer on the austenitic alloy and the variability with which it is removed during post-exposure cleaning. Therefore, the decrease in sound metal specimen thickness was also measured in the case of the stainless steel, and these data are shown in Fig. 6. Because of the uniform corrosion of the Fe-12 Cr-1 MoVW steel, its curve in Fig. 6 was simply generated from weight loss data.

Discussion

As shown by the representative micrographs in Figs. 1, 3, and 4, the attack of type 316 stainless steel was quite extensive and produced a "spongy" surface layer. The "channeling" of liquid metal into the solid resulted in preferential depletion of nickel (and some chromium) and a transformation of the austenite to ferrite in the corrosion zone. The corrosion-induced morphological changes observed in the present experiments are in general agreement with other studies [2-5]. In particular, the x-ray images of corroded type 316 stainless steel shown in Fig. 1 are quite

similar to those of type 316L stainless steel exposed to both static, 600°C [2] and thermally convective, 450°C [4] Pb-17 at. % Li.

In considering the degree of attack shown in the above micrographs, it is reasonable to expect somewhat poor adherence between the porous ferritic layer and the underlying austenitic matrix. Indeed, the studies of Fauvet et al. [4] and Chopra and Smith [5] have shown that this corrosion layer can become detached during the lithium soak cleaning procedure. In the present work, it was found that the layer was removed during cleaning from specimens that had previously experienced more severe corrosion conditions. However, the observations from this study, and also those of Chopra and Smith [5], show that, in general, the ferritic layer on corroded austenitic alloys is not always detached during the process of dissolving the residual lead-lithium. The amount of the layer that is removed appears to depend on the depth of the corrosive attack (and thus, on the severity of the environmental reactions), the morphology of the reaction zone, and the microstructural aspects of the interface between the corroded surface region and the unaltered base metal.

Based on the above observations, the effects of specimen cleaning of austenitic stainless steel corrosion coupons previously exposed to lead-lithium are important considerations in planning a methodology for measuring mass loss kinetics and in evaluating the weight change data. The possibility of removing part or all of the ferritic layer, in addition to the residual lead-lithium, makes it imprudent to reinsert the same specimens into the lead-lithium loop to generate mass loss data as a function of time. If the spongy layer is removed during the cleaning process, reexposure of the

specimen would result in the growth of a new layer and cause more rapid corrosion than if the layer had remained. This is thus somewhat different from many (but not all) lithium loop experiments with austenitic stainless steel, where the corrosion layer is not lost during the process of lithium removal prior to weighing. In the case of lithium, reinsertion of the specimens does not normally expose a fresh austenitic surface. A striking illustration of this effect in lead-lithium is shown in Fig. 7, which compares the weight loss measurements from several coupons exposed for different times to weight loss data obtained from a single type 316 stainless steel specimen that was repeatedly exposed to thermally convective Pb-17 at. % Li, cleaned, weighed, and re-exposed. Note the much higher weight losses for the single specimen curve. Furthermore, the weight loss kinetic behavior was quite different; for the multiple specimen curve, weight loss kinetics were described by

$$w = k_e t^{0.7} \quad (1)$$

where w is weight loss, k_e is an effective rate constant, and t is exposure time, while, in the case of the single specimen data,

$$w = k_1 t \quad (2)$$

where k_1 is the linear rate constant. Presumably, the cleaning procedure removed the surface layer and exposed some fresh, nickel-containing surface each time the specimen was put in the loop so that the initial high corrosion rate noted in the multiple specimen case (and in other austenitic stainless steel-liquid metal systems [6]) was maintained. Therefore, in

order to avoid any such difficulties, different specimens were used for each data point in order to generate the mass loss versus time results shown in Fig. 5. Although the specimen cleaning procedure does not strip any corrosion layer from the Fe-12 Cr-1 MoVW steel specimens, the same coupon exposure methodology was used for these specimens to generate the mass loss kinetics data in a consistent manner.

The data presented in the Results Section clearly indicate that the austenitic stainless steel is much more susceptible to corrosive attack by Pb-17 at. % Li than the Fe-12 Cr-1 MoVW steel. The poorer corrosion resistance of the austenitic stainless steel vis-a-vis the Fe-Cr-Mo alloy was apparent not only in the much greater mass losses, but also by the appearance of the steel surfaces. The relatively uniform dissolution of the Fe-12 Cr-1 MoVW steel was in sharp contrast to the deep, irregular attack of the type 316 stainless steel. The weight loss behavior with time also differs for the two alloys; as shown in Fig. 5, the weight losses of the type 316 stainless steel follow a power curve relationship with exposure time [see Eq. (1)], while those of the Fe-12 Cr-1 MoVW steel are approximately linearly dependent on time such that

$$w = k_1' t \quad (3)$$

where w and t are as defined above and k_1' is the linear rate constant. The differing corrosion morphologies and weight loss kinetics for the two types of alloys are similar to those noted from other Pb-17 at. % Li studies with these steels [5] and also reflect what is typically observed in molten lithium environments (see, for example, ref. 8).

as described elsewhere for type 316 stainless steel in lithium [6], a relationship of the form of Eq. (1) can be interpreted as being indicative of a mixed control process consisting of parabolic and linear terms:

$$w = k_p t^{1/2} + k_l t \quad (4)$$

where the first term dominates at early exposures, when nickel is being dissolved at a relatively high rate, and the second term becomes more important at longer times, when the constituents of the steel are released into the liquid metal in proportion to their starting concentration [9]. A weight loss (or dissolution) rate can then be obtained from long-term data by determination of the slope of the weight loss versus time curve (k_l'). Such a procedure is commonly used for liquid metals when linear kinetics have been established and was used in the present case for $t > 3000$ h to get an approximate rate constant (dissolution rate) of $76 \text{ mg/m}^2 \cdot \text{h}$ for type 316 stainless steel exposed to thermally convective Pb-17 at. % Li at 500°C . This value can then be compared to that determined from the weight loss data for the Fe-12 Cr-1 MoVW steel [k_l' , see Eq. (3)]: $21 \text{ mg/m}^2 \cdot \text{h}$. However, because of the basic difference in corrosion morphologies (see above), such a comparison between the austenitic stainless steel and the Fe-12 Cr-1 MoVW alloy is not a definitive one, and a comparison between the decrease in sound metal thickness of each steel is more appropriate. Assuming linear kinetics for the decrease in sound metal thickness, the appropriate rate constants for type 316 stainless steel and Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li at 500°C are 370 and $24 \text{ } \mu\text{m/year}$, respectively. The difference in these surface recession rates is relatively greater than the

difference in the weight loss rates of type 316 stainless steel and Fe-12 Cr-1 MoVW steel because simple mass loss does not account for the lack of integrity of the porous layer on the austenitic alloy. Such determinations of corrosion losses by thickness changes therefore increase the difference in susceptibility between type 316 stainless steel and Fe-12 Cr-1 MoVW steel relative to that obtained from the weight loss measurements.

Any mechanistic explanation for the type of corrosion layer that forms on the exposed type 316 stainless steel must also be consistent with the observation that the Fe-12 Cr-1 MoVW steel corrodes quite differently. This leads to consideration of a model in which the higher solubility and preferential dissolution of nickel account for the nonuniform nature of the corrosion of type 316 stainless steel. Such a model can be based on the work of Harrison and Wagner [10], who showed that preferential leaching of one component of a solid alloy into a liquid metal can destabilize the solid-liquid interface to produce a highly irregular, porous surface, that is very similar to what is observed in the present case for type 316 stainless steel (see Figs. 3 and 4). If one elemental component is dissolving rapidly, a slight physical or chemical perturbation of the interface between the solid and liquid can develop into large scale nonuniformity of this surface. On the other hand, Harrison and Wagner [10] also showed that when the solubilities of alloy components in the liquid metal do not differ greatly from one another (as in the present case of the Fe-Cr steel), the solid alloy-liquid metal interface should remain macroscopically plane in accordance with our observations for the Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li (see Fig. 2).

Much of the experimental work of Harrison and Wagner [10] involved copper-nickel alloys in molten silver at 1000°C, where copper was preferentially depleted. Although solid state diffusion can play an important role under such conditions, calculations show that, in the present case of preferential leaching of nickel at 500°C, the influence of such diffusion is negligible even at the extended exposure times of this study. Nevertheless, for the case where many thin liquid zones infiltrate the solid alloy (in a somewhat similar, though idealized, manner to the what is observed in the present experiments), the concentration of nickel in these liquid regions can be considered a function of time and distance from the original surface such that liquid phase diffusion is the important factor in growth of the zones. For this case, where the liquid zones are fairly close together, Harrison and Wagner [10] derived an expression relating the depth of penetration of the liquid metal, x , to exposure time:

$$x \approx 2 (KN_{Ni}^{(0)} D_{Ni}^{(l)} t)^{1/2} \quad (5)$$

where K is the distribution coefficient of Ni between the liquid and solid, $N_{Ni}^{(0)}$ is the initial mole fraction of Ni in the alloy, and $D_{Ni}^{(l)}$ is the diffusion coefficient of nickel in the liquid phase. While recognizing that measurements of x from present observations are somewhat inaccurate due to possible removal of some of the corrosion layer during specimen cleaning (see above), it is interesting to note that, when x is measured from micrographs of the cross sections of the appropriate stainless steel specimens, such data follow a similar time dependence to that predicted by Eq. (5):

$$x = At^{0.6}$$

(6)

Such relatively good agreement can serve as additional support for the validity of this model in describing the observed corrosion process.

It is informative to compare the corrosiveness of Pb-17 at. % Li to that of another possible tritium breeding fluid — lithium. Under similar conditions, the general characteristics of the corrosion processes are the same for the two liquid metals. In both environments, austenitic stainless steels form a spongy surface layer and exhibit a power curve dependence of weight loss on time [7,8], while Cr-Mo ferritic steels corrode uniformly and show linear weight loss kinetics [8]. However, although overall similar corrosion processes may occur in both systems, Pb-17 at. % Li is much more aggressive than pure lithium. Weight losses, depths of the ferritic layers on type 316 stainless steel, and dissolution rates are all substantially greater in lead-lithium. Furthermore, while porous ferritic layers form on type 316 stainless steel in both Pb-17 at. % Li and lithium, there are differences in their morphologies. Those induced by lead-lithium appeared to have greater and more extended voidage. The greater aggressiveness of lead-lithium toward ferrous alloys can be expected based on higher solubilities in pure lead and the generally more rapid corrosion kinetics observed in pure lead environments (see for example, ref. 11).

In both lead-lithium and lithium environments, the greater corrosion susceptibility of the type 316 stainless steel relative to the Fe-12 Cr-1 MoVW alloy can be attributed to the presence of nickel in the former steel. Other studies with pure lead [11] and pure lithium [12] have

shown that corrosion of Fe-Cr-Ni alloys increased with increasing concentration of nickel, which has relatively high solubilities in molten lead and lithium relative to chromium and iron. Furthermore, as discussed above, the preferential leaching of nickel can lead to the severe type of corrosive attack observed for type 316 stainless steel.

Summary

Type 316 stainless steel and Fe-12 Cr-1 MoVW steel were exposed in thermal convection loops of Pb-17 at. % Li for greater than 6000 h. Results showed that at 500°C, severe, nonuniform attack of the type 316 stainless steel resulted in the formation of extensive ferritic corrosion layers. In contrast, the Fe-12 Cr-1 MoVW steel exhibited uniform corrosion. The presence of the ferritic layer on the type 316 stainless steel and the effects of specimen cleaning on its integrity were shown to be important considerations in evaluating the data and in comparing corrosion losses for the two types of alloys. By any measure, however, the type 316 stainless steel was much more susceptible than the Fe-Cr-Mo alloy to corrosion by Pb-17 at. % Li. In addition, both alloys were corroded to a greater extent in Pb-17 at. % Li than in lithium under similar exposure conditions.

A model from the literature was suggested as an appropriate explanation for the deeply penetrating, irregular corrosion of the type 316 stainless steel by Pb-17 at. % Li. It was based on the preferential dissolution of nickel and the instability of a slightly irregular surface under such conditions. Such a model explains not only the formation of such a layer and its growth kinetics, but also the contrasting uniform corrosion for Fe-12 Cr-1 MoVW steel.

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Table 1. Wavelength dispersive analysis of the polished cross section of type 316 stainless steel exposed to Pb-17 at. % Li for 6348 h at 500°C

Location	Concentration (wt %)			
	Ni	Fe	Cr	Mo
Outer edge of corrosion zone	3.8	86	7.4	2.5
Middle of corrosion zone	3.7	85	8.3	2.4
Middle of corrosion zone	3.5	83	8.9	2.4
Near edge of internal void	3.3	86	7.5	2.6
Near edge of internal void	3.8	85	7.9	2.2
In corrosion zone next to interface with matrix	3.0	84	7.7	2.5
In matrix next to interface with corrosion zone	13	65	17	2.2
Deeper into uncorroded matrix	13	65	18	2.4

Figure Captions

Fig. 1. Backscattered electron images (BEI) and electron microprobe elemental maps of polished cross section of type 316 stainless steel exposed to thermally convective Pb-17 at. % Li for 2015 h at 500°C. (a) Liquid-corrosion layer interface. (b) Corrosion layer-matrix interface.

Fig. 2. Polished cross section of Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li for 2000 h at 500°C.

Fig. 3. Cross sections of type 316 stainless steel exposed to Pb-17 at. % Li for 2472 h at 500°C. (a) Polished without attempting to remove residual lead-lithium. (b) Cleaned in lithium prior to polishing.

Fig. 4. Scanning electron micrograph of view normal to surface of type 316 stainless steel exposed to Pb-17 at. % Li for 2472 h at 500°C.

Fig. 5. Weight loss versus exposure time for type 316 stainless steel and Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li at 500°C.

Fig. 6. Thickness decrease versus exposure time for type 316 stainless steel and Fe-12 Cr-1 MoVW steel exposed to Pb-17 at. % Li at 500°C. The Fe-12 Cr-1 MoVW steel curve was generated from the weight loss data.

Fig. 7. Weight loss versus exposure time for type 316 stainless steel exposed to Pb-17 at. % Li at 500°C. Circles represent data from individual specimens exposed only once. Squares are data for one specimen that was repeatedly cleaned and reexposed.

a

b

fig. 1

fig. 2

fig. 3