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FERROUS ALLOY METALLURGY — LIQUID LITHIUM CORROSION AND WELDING

Progress Report

MASTER

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ABSTRACT

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The effects of liquid lithium on the corrosion behavior and mechanical integrity of ferrous alloys are being evaluated. Fatigue crack growth, which results due to a high local strain at the tip of a propagating fatigue crack, has been used to evaluate the interaction between liquid lithium and an imposed stress. Fatigue crack growth data on type 304L stainless steel at 700C and 2 1/4Cr-1Mo steel between 500 and 700C show that for all imposed test conditions (i.e. frequency, temperature, and nitrogen content in the lithium) the interaction of lithium with the strain at the crack tip results in enhanced crack growth rates. The enhanced growth rates result from the effects of either enhanced grain boundary penetration or a change in crack propagation mechanism due to liquid metal embrittlement. Auger spectroscopy of grain boundary penetrated specimen show that a lithium-oxygen compound forms at the grain boundary. Mossbauer evaluations of the ferrite layer of corroded type 304 stainless steel are being used to develop a model for weight loss in liquid lithium.

The welding research in progress is directed to understand and characterize the influence of variations of the austenitic weld metal composition on the microstructural and mechanical properties of dissimilar metal weldments. Weldments of 2 1/4Cr-1Mo steel to 316 stainless steel have been investigated for fusion microstructure, thermal expansion impact strength and characterization of specific long time in-service failures. Modification of weld metal microstructures by microalloy additions is being investigated as a concept to improve weld metal properties. The behavior of a strip electrode in a gas metal arc is being investigated to determine the feasibility of gas metal arc weld strip overlay cladding.

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1.0 INTRODUCTION

The results obtained from January 1, 1980 to December 31, 1980 on the project entitled "Ferrous Alloy Metallurgy - Liquid Lithium Corrosion and Welding" are summarized in this progress report. Section 2.0 presents results on the liquid lithium corrosion study and Section 3.0 presents the results on the welding study.

The liquid lithium research has had during the year two main objectives: (1) The identification and understanding of the corrosion products involved in liquid lithium attack. A special effort (section 2.3) to use Auger Secondary electron spectroscopy to identify the corrosion products along penetrated grain boundaries has been applied and has given some information as to the nature of the grain boundary damage. Mossbauer spectroscopy has also been applied (section 2.2) to relate the growth of the ferrite boundary region of corroded austenitic stainless steel to reported weight loss kinetics. The ferrite boundary region serves as a protective barrier to liquid alkali attack and needs to be well understood for future alloy development. (2) A major effort (section 2.1) has been given to the identification of the role deformation processes ahead of a fatigue crack has on the lithium enhanced crack propagation found in both 2 1/4Cr-1Mo steel and type 304L stainless steel. The lithium enhanced crack propagation has been seen in both high and very low nitrogen contents of the liquid lithium suggesting that it is a lithium-mechanical interaction and not related to the nitrogen contamination as has been suggested by other investigators.

The nature of the fracture surface suggest a liquid metal embrittlement phenomena and has promoted our investigating fatigue behavior just above the melting point where liquid metal embrittlement has been reported to be the most severe.

The welding research program, summarized in section 3.0, is primarily concerned with dissimilar metal welding metallurgy with the special emphasis on austenitic welding consumables. Particular interest has been directed to the 2 1/4Cr-1Mo steel to type 316 stainless steel weldments since they represent the classic ferritic to austenitic welding problems. Ferritic to austenitic welding concerns suggest research into both residual stress and influence of weld chemistry on microstructure and properties. One effort (section 3.6) is directed at determining the thermal expansion coefficients as function of effective nickel and chromium contents since there often results large thermal expansion differences and thus residual stresses from these welds. It is anticipated with these results that iso-expansion lines can be drawn on the Schaeffler diagram from which selection of an austenitic welding consumable can be made that gives better thermal expansion compatibility across the joint as well as an acceptable fusion zone ferrite content.

The influence of dilution on the weld metal chemistry, microstructure, and mechanical properties of various austenitic weld metals used to weld 2 1/4 Cr-1Mo steel to austenitic stainless steel has been investigated (section 3.1). Major differences in weld metal properties are observed when comparing dissimilar metal austenitic weld metal to similar austenitic weld metal or parent wrought austenitic stainless steel.

Another effort (section 3.5) has been to carefully characterize the microstructure of a 2 1/4Cr-1Mo steel heat affected zone of a ferritic to austenitic dissimilar metal joint which has experienced an apparent creep failure after 15 years of high temperature service. The carbide morphology, nature, amount, and distribution along the fracture surface is being characterized so that accelerated heat treatments can be made of sufficient 2 1/4Cr-1Mo steel to simulate the specific region of failure in the heat affected zone, a heterogeneous material. With the accelerated heat treatment to simulate a specific heat affected zone microstructure various alloy modifications of 2 1/4Cr-1Mo steel can be studied for their long term creep resistance.

A special effort (section 3.1) is being made to understand the role of microalloy additions to modify weld metal microstructure and reduce hot cracking susceptibility by gettering the lower melting segregating elements. This effort is presently studying an aluminum alloy system but in the future it will be applied to austenitic fusion zones.

Weld overlay cladding results in a dissimilar metal weldment which has all the typical problems but must also produce the proper alloy composition and microstructure to serve its intended function, ie. corrosion, wear, etc. Gas metal arc weld overlay strip cladding is being investigated to allow higher deposition rates with an all position welding process. Most of this year's effort has been to develop strip feeders and investigate the arc phenomena and melting of a strip electrode in an argon arc atmosphere.

2.0 RESULTS ON LIQUID LITHIUM PROGRAM FOR 1980

2.1 FATIGUE CRACK PROPAGATION OF FERROUS ALLOYS IN LIQUID LITHIUM

The testing procedures described previously, (1,2,3) have been utilized in this contract year to more completely evaluate as a function of frequency, temperature, and nitrogen content the fatigue crack propagation characteristics of 2 1/4Cr-1Mo steel and 304L stainless steel in liquid lithium.

Fatigue crack growth rates for 2 1/4Cr-1Mo steel in lithium with a high (2000 ppm) nitrogen content and in argon were determined at 673 and 873K for frequencies of 0.067 to 20 Hz. In addition crack propagation characteristics were analyzed with SEM fractography. At a constant imposed stress intensity factor range, ΔK , fatigue crack growth rates in argon at 673 and 873K decreased with increasing frequency. This behavior is similar to the results reported previously for 773K (1,3), and is summarized in Fig. 1 at a constant stress intensity factor range of 20 MPa M. These data show similar crack propagation characteristics in lithium and argon at 873K. In contrast at 673 and 773K growth rates are similar at low frequencies but diverge at high frequencies, with the growth rate in lithium increasing with an increase in frequency.

The increase in crack growth rate in lithium at 673 and 773K has been shown (4) to result from a crack propagation mechanism mode change from normal plasticity controlled transgranular fatigue in argon (as shown in the SEM fractograph presented in Fig. 2a) to liquid metal embrittlement controlled transgranular cleavage in lithium (as shown in the fractograph presented in Fig. 2b). In

contrast for the frequency range investigated at 873K, SEM analysis showed that the crack propagation mode is not a function of environment.

The data presented above show that the presence of liquid lithium enhances fatigue crack growth rates in 2 1/4Cr-1 Mo steel between 673 and 873K in the frequency range of 0.067 to 20 Hz by two distinct processes: stress enhanced grain boundary penetration at low frequencies and strain rate induced cleavage by liquid metal embrittlement at high frequencies.

The crack propagation characteristics of 2 1/4Cr-1Mo steel were also determined in liquid lithium with a low (< 100 ppm) nitrogen content at 773K at frequencies of 0.67 Hz and 20 Hz. In Fig. 3 the crack growth rate versus frequency curve in high lithium with a high nitrogen (2000 ppm) content (shown previously in Fig. 1), is compared to the growth rates at 0.67 Hz and 20 Hz in lithium with less than 100 ppm N. The similarity between the growth rates in lithium with both a low and high nitrogen content show that the observed embrittlement at high frequencies is a result of the effects of the lithium and not the nitrogen contamination. Current studies are directed at modeling the embrittling process by considering the strain rate induced liquid metal embrittlement model presented previously.

The effects of lithium and frequency on the fatigue crack propagation rate of type 304L stainless steel at 973K have been reported previously (1,5). These results showed that the crack growth rate increased with decreasing frequency and that lithium increased the growth rate at all frequencies in the range of 0.067 to 20 Hz. Optical metallography and SEM fractography have shown the

crack propagation mode to change from intergranular at 0.067 Hz to transgranular at 20 Hz in argon. On the basis of these results it is proposed that strain-enhanced grain boundary penetration at the tip of the propagating fatigue crack enhances crack propagation in lithium.

The nature of lithium penetration near a crack tip under stress was investigated by holding precracked CT specimens (identical to those used in the fatigue studies) at a constant load for 68 hr. in lithium and in argon at 973K. The initial applied ΔK in the argon test was 11.4 MPa $\sqrt{m}$ and that for the lithium test was 10.4 MPa $\sqrt{m}$. The difference in K was the result of different initial crack lengths.

Sections of the sample tested in lithium were examined for grain boundary penetration in unstressed regions as well as at the crack tip. Figure 4 contains optical micrographs from the various points on the sample indicated in Fig. 4. Figure 4a illustrates the penetration observed on the sample surface which was hand ground prior to testing. This penetration is comparable to that for the precrack zone through which the fatigue crack passed at room temperature prior to testing (Fig. 4b). The magnitude of this penetration is very close to the 78 microns predicted for unstressed type 304L stainless steel at N=0.2 wt%, T=973K, and t=68 hr. (The value of 78 microns was calculated from the equation reported by Olson et al.(6)). A marked increase in penetration was observed for the side of the pin hole which was loaded in compression during precracking and testing as shown in Fig. 4c. Sections from machined surfaces which were not stressed during testing showed penetrations very similar to those shown in Figs. 4a

and 4b. Fig. 4c is qualitative evidence of strain-enhanced penetration in type 304L stainless steel similar to that reported for Armco iron (7).

Low magnification micrographs of the constant load samples revealed the expected results as shown in the crack tip micrographs presented in Fig. 5. Dark field illumination accentuates the penetration in these lightly etched samples. Figure 5a shows no grain boundary attack other than a few regions where a second phase, presumably oxide, was present at the grain boundaries of the specimen tested in argon. Figure 5b on the other hand, shows a uniform zone with greater penetration in the region near the tip of the crack which was stressed while submerged in lithium.

The effect of lithium increasing crack propagation is evidenced by three observations:

- 1) equal crack extensions with a lower applied stress in lithium,
- 2) equal crack extension with less deformation (crack tip opening) in lithium, and
- 3) two cracks propagating to approximately the same length in lithium compared to only one major crack in argon.

It is expected that the effect of propagating two cracks simultaneously is to lower the effective ΔK for each crack.

Figure 5b and Fig. 4c contain evidence for strain-enhanced penetration under tensile and compressive loading. The mechanism for this phenomena is not understood but it may be the same as that for Armco iron under creep conditions. It is doubtful that a film-rupture mechanism would apply as plastic deformation in compression would tend to close grain boundaries inhibiting further

Figure 1. Crack growth rate, da/dn , as a function of loading frequency for 2 1/4Cr-1Mo steel tested in lithium at 673, 773, and 873 K.

Figure 2. SEM fractographs of 2 1/4Cr-1Mo steel samples tested in argon (2a) and lithium (2b) at 773K and 20 Hz showing lithium induced cleavage.

Figure 3. Crack growth rate, da/dn , as a function of loading frequency for 2 1/4Cr-1Mo steel tested in high nitrogen lithium (the solid line) and low nitrogen lithium (the individual data points).

(a)

(b)

(c)

(d)

Figure 4. Optical micrographs showing the penetration observed in a specimen held in lithium for 68 hours at a constant load. As shown in (d), (a) is the hand ground surface, (b) is the fatigue precrack zone, and (c) is the side of the pin hole which was loaded during exposure. Etched with 5g FeCl_3 , 2ml HCl , 96ml ethanol.

(a) ARGON

(b) LITHIUM

Figure 5. Dark field micrographs of the crack tips of the specimens held at a constant load of 400 lb (1980 N) in argon (a) and lithium (b). Etched with 5g FeCl_3 , 2ml HCl , 96ml ethanol.

penetration, especially if a corrosion product were formed. It is more likely that deformation enhances diffusion by creating excess vacancies, by producing dislocations for pipe diffusion, or by increasing grain boundary diffusivity due to grain boundary sliding. Current studies are underway to understand the mechanisms involved.

2.2 INVESTIGATION OF THE FERRITE LAYER ON LITHIUM CORRODED STAINLESS STEEL

When dissolution of type 304L stainless steel occurs in liquid lithium, chromium, nickel, and iron are removed from the surface at unequal rates (8). The phase diagram of Fig.6 indicates that although this stainless steel lies in an austenitic phase field, small changes in composition may result in an austenite to ferrite transformation. Thus exposure of an austenitic stainless steel to liquid lithium may result in formation of a ferritic surface layer. This ferrite layer serves as a passive or nearly passive protective barrier for corrosion depending of the nitrogen content of the liquid lithium. The growth rate of the ferrite layer depends on both the austenite/ferrite interface velocity and ferrite/liquid lithium interface reaction kinetics. The latter is a strong function of nitrogen content. A Mossbauer spectroscopy study of the ferrite layer growth phenomena is being conducted at CSM.

Mossbauer spectroscopy is a gamma-ray resonance technique sensitive to phase changes. In particular, the thickness of a ferrite layer on an austenitic substrate may be measured. Analysis of Mossbauer spectra from a series of stainless steel corrosion coupons allows the ferrite layer growth kinetics to be determined.

Several grades of austenitic stainless steel are being evaluated, the emphasis being on type 304L stainless steel. Type 304L stainless steel coupons were evaluated at 1073K, 1123K, and 1173K for nitrogen concentration of both 500ppm and 1500ppm. For each test condition, a series of corrosion coupons are exposed to lithium for times ranging up to 400 hours, allowing weight loss kinetics to be determined. A Mossbauer spectra is then being obtained from each sample, and the ferrite layer thickness is calculated. In this manner, weight loss and ferrite layer growth kinetics are both currently under study.

Long-term capsule tests (9) indicate that the ferrite layer grows at a decreasing rate, eventually reaching a limiting thickness. When it reaches this limiting thickness it is believed that the kinetics change from a parabolic to linear time dependence. Tests of 400 hours run under high-nitrogen conditions (2000ppm) at CSM indicate a ferrite layer thickness of approximately 6 microns. The tests outlined previously have not yet been completed, but it is known that low nitrogen concentrations will result in more rapid ferrite layer and lower weight loss rates.

It is anticipated, when testing is completed and Mossbauer data analyzed, that weight loss and ferrite layer growth kinetics may be correlated. It is known (9) that weight loss rates eventually decrease to very small values during long-term tests. This phenomena may be controlled by the ferrite layer thickness. If for example, the rate-controlling step in dissolution involves diffusion through the ferrite, then an increasing ferrite layer thickness would result in the weight loss rate decreasing with time, as has been experimentally observed.

Figure 6. Iron-nickel-chromium phase diagram at 1373 K. The solid dot represents the nominal composition of type 304L stainless steel.

2.3 CORROSION PRODUCT IDENTIFICATION

Lithium corrosion along the penetrated grain boundaries of 2 1/4Cr-1Mo steel samples has been studied with Auger Electron Spectroscopy on fractured surfaces. The purpose of the investigation was to identify the corrosion product and to determine the chemical bonding of the lithium. The latter is possible since a single lithium atom has only one outer shell electron and cannot undergo an Auger transition by itself. Any lithium Auger transition will require an electron from a neighboring atom, resulting in a different spectrum for each Li-X bond.

Lithium compounds studied to show this Li-X bond dependence were: Li_3N , LiNO_3 , Li_2O , Li_2O_2 , Li_2CO_3 , and Li_2C_2 . Excessive sample charging prevented collecting information about the nitrogen compounds but the other compounds gave two different spectra in the 0-100 eV range. Li_2O , Li_2O_2 , and Li_2CO_3 presented the same low energy spectrum (Fig. 7a), that of an Li-O bond. The Li_2C_2 spectrum (Fig. 7b) shows different peak positions and separations as a result of the Li-C bond. The compounds can be identified by combining the above low energy spectra with the high energy peaks (0-1000 eV). Samples for AES analysis were small tensile bars. All samples were heat treated prior to being placed in the liquid lithium to achieve a microstructure similar to that known to be lithium attacked in the heat affected zone of 2 1/4Cr-1Mo steel weldments. After being annealed and corroded in liquid lithium, the samples were transferred to a protective atmosphere glovebox connected to the spectrometer. The samples were then fractured and inserted into the

spectrometer without being exposed to the atmosphere.

The sample fractures were predominately intergranular in the corroded regions and transgranular through the rest of the sample. Figures 8a and 8b show the low and high energy Auger Electron spectra from a corroded area. The product can be identified from the spectra as having primary lithium-oxide bonds since no carbon (270 eV) or nitrogen (379 eV) peaks are present. Figure 8a shows a Li-O bond spectrum. The iron peaks are due to the lithium-oxide layer being thin enough to allow resolution of the iron Auger electrons.

The results of a series of Auger spectra taken across two samples are shown in Fig. 9a and Fig. 9b. These are plotted as normalized peak heights (the sum of all peaks on a single spectrum equals 100). This is not a concentration, but it does show relative changes in the spectra. The "edge" is the outside of the sample and was not always well defined. The beam size was about 20 microns in diameter.

Both scans show that when the grain boundary is covered by a lithium corrosion product, it can be associated with primary lithium-oxide bonds. Figure 9a shows the presence of calcium, which was not present in the chemical analysis of the steel. The evidence of the calcium is a concern since calcium is a known residual element in high purity lithium. Further work is necessary to clarify the source of the grain boundary calcium for it may influence the purity of lithium required for advanced energy systems.

The AES studies have shown the corrosion product on the grain boundaries to be an oxide associated with lithium. Furthermore it

Figure 7. A comparison of the Auger patterns for Li_2CO_3 (7A) and Li_2C_2 (7B) showing the shift in peak voltage with bonding.

(A)

(B)

Figure 8. The low energy (8A) and high energy (8B) Auger spectra for lithium corroded grain boundaries of 2.1/4Cr-1Mo steel.

(A)

(B)

Figure 9. Correlations of normalized peak heights measured on two samples of 2 1/4Cr-1Mo steel (A&B) with distance from the lithium/metal interface. Measurements were made on the corroded grain boundaries and indicate the variation in corrosion product composition with depth of penetration.

has been established that lithium-carbon bonds do not form readily and that nitrogen is not in the corrosion product (within detection limits of the spectrometer). Work is in progress to study the grain-corrosion product interface using AES depth profiling.

2.4 EFFECTS OF MICROSTRUCTURAL STABILITY ON THE LITHIUM CORROSION OF 2 1/4Cr-1Mo STEEL

The recent study to more completely describe the interactions between the various carbide structures of 2 1/4Cr-1Mo steel and the liquid lithium corrosion mechanism was concluded. In a previous study(10), the heat affected zones of spot welded coupons were determined to be the most susceptible regions for corrosion by lithium. Therefore, additional work was done to simulate the region by using a heat treatment. A heat treatment was developed and was reported in the 1979 Progress report(1).

Simulated weldment coupons were exposed to liquid lithium - 17.6 wt% lead in an attempt to generate information leading to an understanding of the corrosion mechanisms responsible for the enhanced corrosion associated with the heat affected zones.

Penetration kinetics for various temperatures are shown in Fig. 10. In Fig. 10, it can be seen that the penetration curves for intermediate temperatures exhibited a change in slope, while both higher and lower temperature corrosion data yielded straight lines. The change in slope apparently corresponds to a change in corrosion mechanism as well as a change in carbide morphology (epsilon carbide transforming to cementite, Fig. 11). It was concluded that at lower

Figure 10. Corrosion of simulated weldments (oil quenched from 1300C) as a function of temperature and the square root of time.

Figure 11. Comparison of the change in corrosion rate with the conversion of epsilon carbide to cementite in quenched 2 1/4 Cr - 1 Mo steel.

Figure 12. Arrhenius plot of the corrosion rate constant (penetration = $(kt)^{1/2}$) as a function of temperature for simulated weldments (oil quenched from 1300C).

temperatures the change in slope would have appeared if the tests were conducted to longer times and that at higher temperatures the transformation occurred at very early test times and did not indicate any mechanism change.

The slopes of the curves in Fig. 10 were measured and the corresponding rate constants were plotted as a function of temperature as seen in Fig. 12. The initial corrosion rate was controlled by a mechanism with an activation energy of 5.3 kcal/mole. The rate controlling step in the secondary corrosion process had an activation energy of 28 kcal/mole.

In performing the study, the assumption was made that lead did not affect the corrosion mechanism or rate controlling step. This assumption is presently being tested. It should be noted that prior heat treatment and austenitizing time had a profound effect on the corrosion process.

Present work is directed towards an evaluation the effect of lead in lithium on the corrosion mechanism with special concern on embrittlement phenomena.

2.5 RELATED LIQUID METAL PROGRAM

In conjunction with work described above, an additional program which is related to reactive metal corrosion is being performed at the Colorado School of Mines.

Liquid rubidium corrosion of alloy A-286, and types 310 and 304 stainless steels are being investigated. The results of these efforts are of interest to the designers concerned with the

containment of Kr-85 gas (nuclear waste) during high temperature processing and gas bottle storage. Kr-85 decays with a beta emission to become rubidium.

The high temperature corrosion tests were performed in test temperature range of 750 to 930K in liquid rubidium which was covered with Argon gas of various high oxygen contamination levels. The oxygen levels were chosen to best represent present krypton storage gas.

The nature of the rubidium attack has been found to exhibit multiple layer corrosion products common to very high temperature atmospheric corrosion. This is realistic considering the oxygen levels of the liquid rubidium. Besides the evidence of general attack on the surface there is also evidence of grain boundary penetration which in a short time far exceeds the general attack. Grain boundary penetration destroys the integrity of these containment materials.

The present effort is to establish kinetic relationships as function of oxygen content of the krypton gas, time, and temperature for both the advancement of the grain boundary penetration and the growth of the various corrosion product layers. The parabolic rate kinetics have been found to best fit our results and we are reporting our work for each of the stainless steels in the following form:

$$x = [k_o [o] \exp(-Q/kt) (t-t_o)]^{1/2}$$

where:

x = penetration or product layer thickness

k_o = rate constant

[O] = is oxygen content in the krypton gas

n = apparent order of reaction

Q = apparent activation energy; in this case more of a temperature coefficient than a mechanistic characterization

t_0 = delay time prior to attack

t = time in liquid rubidium

T = absolute temperature

It is anticipated that these expressions of corrosion damage will allow designers through extrapolation to lower temperatures predict the service lives of various gas storage bottle materials. The temperatures used in testing were consistent with the operating temperature used in processing of the krypton gas and data is useful in the selection of materials for process equipment.

It is interesting that earlier work with low oxygen contaminated rubidium suggested no major corrosion problems and probably represents the more classical characteristics of liquid reactive metals. The high oxygen contents of the liquid rubidium converted the liquid metal attack into an apparent high temperature oxidation problem. It appears that the liquid rubidium removes passive oxide protective films and assists in the delivery of oxygen to the corroding stainless steel-rubidium interface. The evidence that oxidation of chromium is involved can be seen in the Fe-Ni-Cr ternary phase diagram Fig.13 where the chromium loss is apparent by comparing the original alloy composition with the composition of the corroded stainless steel surface region. If the traditional nickel leaching of stainless steel were the dominant mechanism the arrows would indicate a higher nickel loss in alloy A-286 and type 310 stainless steel. It is anticipated that we will have completed our efforts in rubidium corrosion within the next 6 months.

Figure 13. Fe-Ni-Cr ternary diagram showing base metal and corroded surface region compositions.

3.0 RESULTS OF WELDING PROGRAM FOR 1980

3.1 CONTROL OF FUSION ZONE MICROSTRUCTURE

A previous experimental study has shown that the presence of sodium in an aluminum-silicon eutectic alloy base metal will modify the fusion zone microstructure in an autogenous gas-tungsten-arc (GTA) weldment (1,11,12,13,14). This modification consists of a change in morphology of eutectic silicon crystals from a plate shape to a cylindrical shape. A continuing study is now underway to determine if the addition of sodium to an Al-Si eutectic filler wire will have the same modification effect in a gas-metal-arc (GMA) weldment. The question at issue is whether or not sodium will transfer across the arc, from the filler wire to the weld pool.

Filler wire is being fabricated at CSM by chill-casting a 4047 aluminum alloy treated with sodium, into the form of 25.4 mm diameter ingots. These ingots are then rod-rolled to a diameter of 5 mm. (A set of grooved rod-rolls were recently purchased for this purpose.) The 5 mm rod is then reduced to 1.5 mm filler wire using swaging and drawing techniques. A GMA weldment will be made on 1100 aluminum plate using this sodium containing filler wire. Metallographic and micro-tensile specimens will be machined from the fusion zone for analysis. Welds will also be made using sodium free filler wire to provide a basis for comparison. Filler wire treated with strontium (an element found to produce an effect similar to sodium) (15) will also be tested.

In a recent review of potential applications for fusion zone microstructure control (16), it was suggested that the hot tearing

tendency observed in many aluminum-magnesium (Al-Mg) alloy weldments may be reduced by controlling the relative amount of impurities present. During fusion zone solidification the partitioning of magnesium, together with the major impurities iron and silicon, determines the composition of the last liquid to solidify. In the generalized theory of hot tearing developed by Borland(17), hot tearing phenomena is related to the interfacial energy between the solidified crystals and the last liquid to solidify (ie: an inner-crystalline liquid film). If the last liquid to solidify will wet these crystals (ie: low interfacial energy), then the liquid film tends to be continuous and any hot tearing that occurs (caused by shrinkage strains) will be "self-healed". If the last liquid to solidify does not wet (ie: high interfacial energy), then solid-solid bonding between adjacent crystals is allowed to occur and hot tearing is again avoided. It is at intermediate degrees of wettability that hot tearing becomes a problem (ie: non-continuous films and small amounts of solid-solid bonding).

It has long been known that the hot tearing susceptibility of Al-Mg alloys varies with magnesium content (18). However, it was just recently discovered that iron and silicon impurities also influence hot tearing susceptibility for any given magnesium content (19).

Research work on this subject is currently in progress at CSM with the intention of clearly defining the mechanism of fusion zone hot tearing in the Al-Mg-Fe-Si alloy system. High purity alloys of aluminum with controlled amounts of magnesium, iron, and silicon are being tested in a vareststraint fixture (discussed in section 3.4) to determine relative susceptibility to hot tearing. A computer

simulated model for solute redistribution is being constructed to predict the composition of the inner-crystalline liquid film. Computer results will be compared with optical measurements taken from varestraint welds. Interfacial energy, as a function of liquid film composition, will also be considered.

The ultimate goal of this work will be to reduce the hot tearing susceptibility of commercial Al-Mg alloys by means of micro-alloy control. This might be accomplished by one of four methods:

- 1) Directly control the relative amounts of micro-impurities such as Fe and Si.
- 2) Indirectly control the relative amounts of micro-impurities (Fe and Si) using micro-alloy additions to form high temperature Fe/Si compounds.
- 3) Increase (or decrease) liquid film interfacial energy using micro-alloy additions.
- 4) Control the amount of other micro-impurities (Cu,Cr,Zn) that might influence liquid film interfacial energy.

3.2 EVALUATION OF MICROSTRUCTURE IN A DUPLEX STAINLESS STEEL WELDMENT

In an on-going study to characterize the microstructural and mechanical properties of dissimilar metal weldments (type 316 stainless steel to 2 1/4Cr-1Mo steel) (1,20) it is necessary to analyze a duplex (austenite + ferrite) fusion zone microstructure. Advanced metallographic techniques are being refined at CSM to apply quantitative optical microscopy to austenitic weld metal. A duplex microstructure resulting from using a type 312 stainless steel filler to make a dissimilar metal weldment with a two pass gas-metal-arc process was used to illustrate our capability. Using a dilution calculation method discussed previously (1), the composition of the fusion zone (second pass) can be plotted on the Schaeffler diagram as shown in Fig. 14.

To analyze the duplex microstructure, use was made of a color depositional etch developed by Beraha and Shpigler(21). This etch consisted of:

20 g ammonium bifluoride

0.5 g potassium metabisulfite

100 ml distilled water

immersion time: 105 seconds at room temp.

Before color etching, however, the specimen was given a light electrolytic "pre-etch" in 10% oxalic acid to provide relief and enhance optical resolution. The resulting microstructure is shown

Figure 14. Schaeffler diagram showing the effect of composition change due to weld dilution of 2 1/4 - 1 Mo steel and 316 stainless steel base metal using 312 stainless steel filler metal.

5 μ

Figure 15. Duplex fusion zone microstructure (austenite and ferrite) of dissimilar metal weldment joined with 312 stainless steel filler metal.

Figure 16. A comparison of a normal optical micrograph (A) of 312 stainless steel weld metal with the corresponding micrograph where the delta ferrite is enhanced with a magnetic fluid. (B)

in Figure 15. Note that the dendritic crystal (green) is a primary delta-ferrite dendrite that has undergone a phase transformation to form austenite. The inner spine of this dendrite should have a high concentration of chromium and is the region of retained delta-ferrite (white), as shown by Lippold and Savage (22). The inner-dendritic region (yellow) is a transformed eutectic.

The location of the retained delta-ferrite was confirmed using a magnetic etching technique developed by Gray (23). This involved placing a drop of ferrofluid (iron filings suspended in water) between a glass cover-slip and the specimen followed by oil immersion optical microscopy. When a magnetic field is imposed on the specimen, the iron filing migrate to the ferromagnetic delta-ferrite as shown in Figure 16. Note the outline of the dendritic spine, as was observed in Figure 15.

It is essential to have accurate quantative measures of the specific austenitic weld metal constituents if the weld metal behavior is to be understood.

3.3 MECHANICAL PROPERTIES OF AUSTENITIC FUSION ZONES IN DISSIMILAR METAL WELDMENTS

The influence of the obvious variations in dissimilar metal fusion zone compositions on the mechanical properties of the weld metal was investigated to determine the sensitivity of the mechanical properties on the fusion zone composition. To properly select, based on analytical procedures, austenitic filler metals for dissimilar metal welding, it is essential that we must understand

quantitatively the following relationships:

- (a) Consumable and base metal chemistries to fusion zone chemistries.
- (b) Fusion zone chemistry to fusion zone microstructure.
- (c) Fusion zone microstructure to mechanical, physical, and chemical (i.e. corrosion) behavior.

In this investigation six different filler materials (five austenitic wires and one ferritic wire) were used in GMA multi-pass welding of 2 1/4Cr-1Mo steel to type 316 stainless steel. The multi-pass welding allowed us to investigate two different dilutions on fusion zone characteristics for each of the six filler metals. Also, some of the welds were post-weld heat treated using the code specified stress relief heat treatment of one hour at 963 K. The processing histories summarized above produced a test matrix with 24 variations in weld metal composition and microstructure.

The welds have been tested as a function of temperature using standard charpy impact testing techniques and the impact data are currently being correlated with microstructure. As an example of the importance of understanding microstructural variations in dissimilar metal weldments consider the temperature dependence of the charpy impact energy data presented in Fig. 17. This figure presents the impact data, in both the as welded and stress relieved condition, of the fusion zone obtained in a dissimilar metal weldment of type 316 stainless steel to 2 1/4Cr-1Mo steel with a type 312 stainless steel filler wire. Also shown for comparison are the corresponding data for wrought type 303 stainless steel. This figure illustrates that the toughness of the fusion zone is significantly more temperature dependant than typical wrought

Figure 17. A comparison of the charpy impact energy versus temperature curve of 312 weld metal both in the as-welded condition and stress relieved with typical data for wrought stainless steel.

austenitic stainless steels. This difference between weld metal and wrought base metal is still being evaluated. Initial SEM studies have shown, even for the weld metal, that ductile rupture mechanisms exist throughout the temperature range in the as-welded material. Further evaluation of the microstructural constituents responsible for the apparent ductile-to-brittle behavior, as exhibited by the impact energy data, are clearly necessary.

To date this study has increased our ability to understand and evaluate the overall behavior of dissimilar metal weldments. The results of this investigation have assisted in the design of new experiments to understand the relationships between chemistry, microstructure, and properties.

3.4 NEW DEVELOPMENTS IN MECHANICAL TESTING OF WELDMENTS

In several portions of this study the importance of understanding the relationships between weld metal microstructures and mechanical properties have been emphasized. A welded joint is best described as a heterogeneous material with variations in microstructure which correspondingly result in variations in strength and toughness. Historically, standard mechanical tests originally designed for homogeneous materials, are applied to evaluate the mechanical properties as a function of position across the weldments. For example, the toughness is evaluated by obtaining a series of fracture samples with notches machined at various locations which correspond to different microstructures in the joint. An alternate method of performing the same type of analysis is to impose a constant strain across a weld bead by forming the

welded plate around a mandrel with a known radius. As all elements of the heterogeneous microstructure experience the same strain, failure will occur in the region of the bead with the minimum toughness.

To utilize the concepts summarized above, a varestraint test system (24) has been constructed as part of an associated test program. The varestraint system applies a known strain to a plate during welding and the sensitivity to hot cracking in the weld bead is evaluated. This system, which is attached to an atmosphere control system, is available for studies of the effects of microalloy additions to the weld metal of similar and dissimilar metal joints. Evaluation of other methods to study the mechanical properties of welded joints are ongoing.

3.5 ANALYSIS OF DISSIMILAR METAL JOINTS FROM FOSSIL FUEL POWER PLANTS

The study of welded joints of 2 1/4Cr-1Mo steel to austenitic stainless steel which have been removed from superheaters in fossil fuel power generating plants has continued. This investigation is essential to our understanding of the service failure modes associated with the use of austenitic consumables so that we can design the proper experiments to obtain the metallurgical fundamental involved. Of the 5 original welds received for evaluation (1) a SA 213-T22 (2 1/4Cr-1Mo steel) steam tube joined to a SA 213-347 stainless steel tube with type 310-Cb stainless steel filler wire was chosen for microstructural characterization. This

weld has been in service for approximately 20 years and has experienced 342 thermal cycles from room temperature to 866K. As shown previously (1) this weld exhibits an oxide notch adjacent to the weld metal and internal damage in the ferritic steel not associated with the oxide notch.

The current investigation involves microstructural characterization of the ferritic material associated with the internal cracking as well as an evaluation of the cracking mechanisms. Of particular emphasis will be a correlation of the internal cracking with the presence of the oxide notch as several (25,26) recent studies have attributed the failure of dissimilar metal weldments to the nucleation and propagation of the surface oxide notch. These data will be used in future work to aid in the development of microstructures for simulating service failures.

The mode of the internal cracking is shown in the montage of optical micrographs shown in Fig. 18. This figure shows that the cracking is primarily intergranular, indicative of a high temperature creep-rupture failure. The orientation of the cracks indicate that the maximum tensile axis was at an angle to the axis of the tube. This orientation supports the theory that one of the causes of weld failure is due to the stresses induced by the effects of the thermal expansion mismatch between the stainless steel and the ferritic base metal.

The surfaces of the internal cracks were investigated as shown in the overview presented in Fig. 19 with SEM on a sample which had been fractured at 77K along the weld metal to ferrite base metal interface to reveal the surface of the internal cracks. In Fig. 20 higher magnifications of the fracture surface of the previously

uncracked base metal (Fig. 20a), the fracture surface of the surface notch (Fig 20b), and the fracture surface of the internal crack (Fig. 20c) are presented. Fig. 20a indicates that at 77K the previously cracked base metal failed by cleavage. A comparison of Figs. 20b and 20c show similar fracture surface morphologies indicative of high temperature creep-rupture failures but that no oxide was present in conjunction with the internal cracks. These observations further support the contention that the internal cracking resulted from the induced stresses between the weld metal and the base metal, and not from an extension of the surface oxide notch or from the welding process.

The fine microstructure which developed in the ferrite with exposure in service, is being evaluated with TEM of wafers of the ferrite cut at various distances from the fusion line. As an example Fig. 21 shows the TEM structure of the 2 1/4Cr-1Mo steel at a position which was unaffected by the welding operation. This figure shows the presence of blocky type carbides decorating both the grain boundaries and subgrain boundaries in the base metal. The carbides have been tentatively identified as $M_{23}C_6$. The evaluation of the carbide structure as a function of position in the heat affected zone is currently under study. The result of this study will provide the background information necessary to design laboratory experiments to simulate in-service operating conditions.

Figure 18. High magnification montage of internal cracking in dissimilar metal weld demonstrating creep-rupture like failure mode.

Figure 19. Low magnification SEM of exposed crack surface showing three areas of interest: Uncracked, surface cracked and internal cracked regions.

Figure 20. High magnification SEM of a) uncracked region showing cleaned morphology b) surface cracked region highly oxidized c) internal cracked region with no oxides.

Figure 21. TEM of base metal 2 1/4Cr-1Mo steel showing carbides developed around grain boundaries and subgrain structures.

3.6 THE EFFECTS OF CHROMIUM AND NICKEL ON THE THERMAL EXPANSION CHARACTERISITICS OF AUSTENITIC STAINLESS STEEL WELD FUSION ZONES

The thermal expansion characterisitics of filler metals becomes important when welds are being made using materials that have different physical or metallurgical properties. This is often the case for welded joints involving stainless steels because of their high and variable thermal expansion properties. If the thermal expansion coefficients (TEC) are known for the base and filler metals then stress analysis techniques can be applied to estimate the thermally induced stresses. Unfortunately, it is difficult to find TEC for stainless steel alloys in the literature, and there is considerable scatter in what data that does exist. Therefore, a systematic study of the TEC of stainless steel weld metal alloys is being conducted. Since it is expected that the amount of retained delta ferrite as well as the composition influences the TEC of austenitic-ferritic stainless steels, an attempt is being made to develop a relationship between delta ferrite and the TEC.

Stainless steel welds are being made with varying chromium and nickel contents and the TEC of each alloy is measured using push rod dilatometry. Since the limited number of stainless steel filler metals was not suitable to produce enough different welds for this study, a weld alloying method was developed. Originally the stainless steel welds were to be made using a submerged arc welding process and the alloying was to be done by adding chromium and nickel powder to the welding flux. This alloying method proved to be less accurate than desired and a second alloying method was developed that has proven to be more consistent. The new alloying

method consists of a three step procedure. First, a gas metal arc weld is deposited on a type 316 stainless steel base plate using a stainless steel filler metal. One of three different deposition rates is used. Next, the weld bead crown is machined off leaving a strip of alloying filler metal in the type 316 stainless steel base plate. Finally, a gas tungsten arc weld pass is used to remelt the filler metal strip and the base metal together. Combining different gas tungsten arc dilution passes with the three different gas metal arc deposition rates results in seven different alloys for each filler metal. These alloys contain approximately 10,15,20,30,40,60 and 90% filler metal.

The three step weld alloying process has been performed on types 308,309 and 310 stainless steel filler metals. Metallographic, chemical and dilatometric specimens are being removed from each weld. Chemical analysis is being performed by Republic Steel Corporation. Dilatometry is performed on these welds once the samples have been machined. Presently the dilatometer has been modified for testing in an inert gas rather than in a vacuum as it is currently being used. Welds will also be made using types 410, 330 and 312 stainless steel filler metals to complete the test matrix.

A computer aided data acquisition system has been set up to digitize and analyze the thermal expansion curves recorded from the dilatometer. Data reduction by the computer results in a time savings and provides more accurate measurements of the TEC than if they were reduced by hand calculations. The final results of this investigation will produce the iso-TEC lines on a Schaeffler Diagram. We are exercising extreme care in obtaining data since we

realize that these will be extremely useful to future mechanical research and design efforts.

3.7 GAS METAL ARC CLADDING AS A POTENTIAL PROCESS

One type of dissimilar metal weld which will be used more frequently is the weld overlay cladding of a corrosion resistant austenitic material on to a pressure vessel structural steel. Weld overlay cladding represents a concept of using the proper material in only the amounts necessary to obtain the benefits of its particular properties. Cladding will become even more valuable in engineering design with the increasing problems of availability of strategic metals.

Austenitic weld overlay cladding is being performed by a variety of different arc welding processes when the welding consumable is a wire. However, only the submerged arc welding process has been used to deposit welding strip. Strip cladding has major advantages in increasing the deposition rate but the submerged arc welding process is limited to only flat position welding which makes it very awkward with extremely large structures or in field welding repair situations.

We have made major efforts to understand the behavior of the metal strip consumable electrode in an argon and argon with 2 to 5% oxygen arc so that weld overlay strip cladding can be achieved by the gas metal arc process. The GMA process can be used in all positions and does not need a flux. The effort to date has involved the building of three different prototype strip feed heads and

torches with various approaches for the introduction of the protective and plasma gas. Each prototype was built based on the information from the use of the earlier version.

We have successfully weld overlay cladded with a one inch strip electrode. AISI 1090 strip (0.5 mm thick) has been used in these initial developments. The welding has to be performed using short circuit transfer since the other transfer modes promote the arc to melt selective portions of the advancing strip edge. The short circuit transfer is usually a lower heat input situation which keeps penetration and thus dilution to a minimum.

We are presently constructing our fourth prototype which is going to give a much better protective atmosphere around the arc and reduce the excessive splattering which has been observed. It is anticipated that the welding equipment requirement will be achieved early next year. When GMA weld overlay strip cladding has been demonstrated as a process, we will start using stainless steel strip as the electrode consumable and perform metallurgical studies as to the resulting microstructures and properties. The stainless steel strip will be deposited on 2 1/4Cr-1Mo steel. The distortion and residual stress concerns will also be investigated.

3.8 COOPERATIVE RESEARCH PROJECT WITH ROCKY FLATS

An investigation (M.S. program of Robert Okagawa) of the influence of nitrogen on the microstructure and properties of stainless steel is in progress. The nitrogen is introduced into the weld metal by systematically altering the plasma gas of a gas

tungsten welding arc in a high pressure chamber. Hyperbaric GMA welding was performed allowing analysis of nitrogen effects as a function of both gas composition and pressure (up to 5 bar pressure was used). Variation in amount and morphology of delta ferrite as function of nitrogen content is being determined. A paper is in preparation. This work is also of interest to hyperbaric underwater welders and represents welding at 150 feet of water.

The influence of capillarity on the stainless steel weld pool morphology is being studied (Ph.D. program of J.R. Roper). The role of specific micro additions to the weld pool is being related to changes in measured interfacial tensions and the flow behavior of the weld pool. The Marangoni effect (27) has been explored, as a major influence on the weld bead morphology problem (i.e. lack of penetration) observed in gas tungsten arc welding a highly refining (electroslag refined) stainless steel alloys. Pendant drop surface tension measuring technique is being used. High speed photography of the weld pool motion is being applied during specific additions to the weld pool.

These projects are being performed at and supported by Rocky Flats Forming R&D.

4.0 REFERENCES

1. D.L. Olson and D.K. Matlock; "Ferrous Alloy Metallurgy-Liquid Lithium Corrosion and Welding", DOE Progress Report, No. C00-2313-9, 1979.
2. D.L. Hammon, S.K. DeWeese, D.K. Matlock, and D.L. Olson; Closed Loop, 1979, Vol.9, No.2, p.3-11.
3. D.L. Hammon; Master of Science Thesis, No. T2380, Colorado School of Mines, Golden, CO, 1980.
4. R.E. Spencer, S.K. DeWeese, D.K. Matlock, and D.L. Olson; Met. Trans.A, 1980, Vol.11A, p.1758-1761.
5. D.K. Matlock, R.E. Spencer, D.L. Hammon, T.A. Whipple, and D.L. Olson; Proceedings of Second International Conference on Liquid Metal Technology in Energy Production, ANS-AIME, Richland, Washington, April 21-24, 1980.
6. D.L. Olson, G.N. Reser, and D.K. Matlock; Corrosion, 1980, Vol.36, p.140-144.
7. T.A. Whipple D.L. Olson, W.L. Bradley, and D.K. Matlock; Nucl. Tech., 1978, Vol.39, p.75-83.
8. N.M. Beskorovainyi, V.K. Ivanov, and M.T. Zvev; High Purity Metals and Alloys-Fabrication, Properties and Testing, 1967, No.5, 1967.
9. P.F. Tortorelli, J.H. Devan, and J.E. Selle; Proceedings of Second International Conference on Liquid Metal Technology in Energy Production, ANS-AIME, Richland, Washington, April 21-24, 1980.
10. T. Anderson, Master of Science Thesis, No.T2372, Colorado School of Mines, Golden,CO, 1980.
11. C. Cross, D.L. Olson, and D.K. Matlock; "Modification of Eutectic Fusion Zone Microstructure", presented at DOE-BES Meeting, Sept. 1979, Oak Ridge, TN.
12. C. Cross, Master of Science Thesis, No.T2290, Colorado School of Mines, Golden, CO., 1979.
13. C. Cross and D.L. Olson; "Modification of Eutectic Fusion Zone Microstructure", presented at TMS-AIME Annual Meeting, Feb. 1980.
14. C. Cross and D.L. Olson; "Modification of Eutectic Fusion Zone

Microstructure", to be presented at AWS Annual Meeting, April 1981.

15. P.D. Hess and E.V. Blackmun; AFS Trans., 1975, Vol. 84, p.87.
16. C.Cross and D.L. Olson; "The Role of Micro-Alloy Additions in the Control of Fusion Zone Microstructure", presented at ASM Annual Meeting, Oct. 1980.
17. J.C. Borland; British Welding Journal, 1960, Vol.7, p. 508.
18. J.D. Dowd, Welding Journal, 1952, Vol.51, p. 448s.
19. B. Evancho; Alcon, private communication, 1979.
20. D.L. Olson and D.K. Matlock; "Dissimilar Metal Welding Research", presented at DOE-BES Meeting, Sept. 1980, Berkley, CA.
21. J.E. Beraka and B. Shpigler, Color Metallography, American Society for Metals, 1977.
22. J.C. Lippold and W.F. Savage; Welding Journal, 1979, Vol.58, p.3623.
23. R.J. Gray, Metallographic Specimen Preparation, ed. J.L. McCall and W.M. Mueller, Plenum Publishing Corp., 1973, p.155.
24. W.F. Savage and C.D. Lundin; Welding Journal, 1965, Vol.11, p.433s-442s.
25. J.T. Tucker, Jr., and F. Eberle; Welding Journal, 1956, Vol. 35, No.11, p.529s-540s.
26. R.W. Emerson, R.W. Jackson, and C.A. Dauber; Welding Journal, 1962, Vol.42, No.9, p.385-393.
27. L.E. Scriver and C.V. Sternling; Fluid Mechanics, 1964, Vol.19, p.321.

5.0 SUMMARY OF RESULTS FOR THE CURRENT CONTRACT YEAR

1. More substantial information as to fatigue crack propagation in type 304 stainless steel and 2 1/4Cr-1Mo steel in liquid lithium has been obtained.
2. Lithium enhanced crack propagation is not a strong function of the nitrogen content of the liquid lithium.
3. Lithium enhanced crack propagation is related to the nature of the deformation process in the vicinity of the crack tip.
4. Fatigue crack propagation in 2 1/4Cr-1Mo steel is enhanced by liquid metal embrittlement induced cleavage at high loading frequencies.
5. The primary bonding associated with corrosion products along penetrated grain boundaries of 2 1/4Cr-1Mo steel has been determined by AES to be a lithium-oxygen bond with no evidence of lithium-nitrogen or lithium-carbon bonds.
6. Ferrite boundary layer thickness measurements of lithium corroded stainless steel are being made by Mossbauer spectroscopy and the measurements will be used to determine the role of ferrite growth on the weight loss kinetics.
7. Vareststraint equipment has been built to assist in the investigation to determine which microalloy addition can increase hot cracking resistance of a weld metal. We are currently studying aluminum alloys.
8. Welding wire drawing capability is being added to our program to

allow us to produce small amounts of microalloyed wire for experimental purposes.

9. An in-service dissimilar metal (2 1/4Cr-1Mo steel to stainless steel) failure has been thoroughly analyzed to determine the specific nature of the microstructure associated with the crack. The carbide structures at the failure (which occurred after 20 years of service) are being characterized to assist in establishing the proper accelerated heat treatment necessary to generate testing specimen material with a similar microstructure.
10. The dilatometer has been modified and is being utilized to determine thermal expansion measurements of austenitic weld metal.
11. Quantitative metallographic and microscopic techniques have been developed for austenitic weld metal.
12. Apparent differences in the mechanical properties between the austenitic fusion zones obtained from dissimilar metal welds and the austenitic fusion zones obtained from similar metal welds have been found. These observations indicate that dilution may introduce detrimental elements to the weld metal.
13. The behavior of a strip electrode in a gas metal arc is being investigated as a first step to produce gas metal arc weld strip overlay cladding.

6.0 SUMMARY OF PROFESSIONAL ACTIVITIES IN 1979.

6.1 Summary of Published Manuscripts

1. D.K. Matlock, R.E. Spencer, D.L. Hammon, T.A. Whipple, and D.L. Olson, "Interrelationship Between Corrosion and Deformation Processes of Materials in Liquid Lithium", proceedings of Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
2. T.L. Anderson, N.J. Hoffman, J.B. Lumsden, G.R. Edwards, "Intergranular Penetration of 2 1/4Cr-1Mo Steel Weldments in a Lithium-Lead Liquid", proceedings of Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
3. F.D. Schowengerdt and J.S. Forrest, "Auger Electron Spectra of Liquid Compounds", proceedings of Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
4. R.E. Spencer, S.K. DeWeese, D.K. Matlock, and D.L. Olson, "Fatigue Crack Propagation Characteristics of 2 1/4Cr-1Mo Steel in Liquid Lithium at 773K", Metallurgical Transactions, Vol. 11A, 1980, pp. 1758-1761.

6.2 Summary of Manuscripts in Preparation or Review

1. W.A. Averill, D.L. Olson, D.K. Matlock, and G.R. Edwards, "Lithium Reactivity and Containment", proceedings of First International Conference on Aluminum-Lithium Alloys, ed. by T.H. Sanders, Jr. and E.A. Starke, Jr., to be published by TMS-AIME, Warrendale, Pennsylvania, 1980.
2. D.L. Olson and D.K. Matlock, "Diamond Cubic Phase of Lead", accepted for publication in Journal of Physics and Chemistry of Solids.
3. D.L. Olson, P. Steinmeyer, D.K. Matlock, and G.R. Edwards, "Corrosion Phenomena in Molten Lithium", accepted for publication in Reviews on Coatings and Corrosion, an International Quarterly Review.
4. T.R. Pinchback, J.R. Winkel, D.K. Matlock, D.L. Olson, "On the Nature of Liquid Rubidium Attack", accepted for publication in Nuclear Technology.
5. C.E. Cross and D.L. Olson, "Modification of Eutectic Fusion

Zone Microstructure", in preparation for submission to The Welding Journal.

6. J.W. Elmer, D.L. Olson, and D.K. Matlock, "Thermal Expansion Characteristics of Austenitic Stainless Steel Filler Metals", in preparation for submission to The Welding Journal.
7. D.L. Hammon and D.K. Matlock, "The Effects of Lithium on Fatigue Crack Propagation in Iron Base Alloys", in preparation for submission to the ASM Energy Journal.
8. P. Steinmeyer, D. Williamson, and D.L. Olson, "Corrosion Kinetics of 304L Stainless Steel in Liquid Lithium as Observed by Mossbauer Spectroscopy", in preparation for submission to the ASM Energy Journal.

6.3 Oral Presentations, Conference and Workshop Activities

1. D.L. Hammon, D.L. Olson, and D.K. Matlock, "Fatigue Crack Propagation in Liquid Lithium at Elevated Temperatures", presented at 109th AIME Annual Meeting, Las Vegas, Nevada, February, 1980.
2. C.E. Cross and D.L. Olson, "Modifications of Eutectic Fusion Zone Microstructure", presented at 109th AIME Annual Meeting, Las Vegas, Nevada, February, 1980.
3. D.K. Matlock, R.E. Spencer, D.L. Hammon, T.A. Whipple, and D.L. Olson, "Interrelationship Between Corrosion and Deformation Processes of Materials in Liquid Lithium", presented at Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
4. T.L. Anderson, N.J. Hoffman, J.B. Lumsden, G.R. Edwards, "Intergranular Penetration of 2 1/4Cr-1Mo Steel Weldments in a Lithium-Lead Liquid", presented at Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
5. F.D. Schowengerdt and J.S. Forrest, "Auger Electron Spectra of Liquid Compounds", presented at Second International Conference on Liquid Metal Technology in Energy Production, Richland, Washington, April 20-24, 1980.
6. R.E. Spencer, D.K. Matlock, and D.L. Olson, "An Evaluation of a Lithium Pump Failure", presented at Lithium Users Workshop, Richland, Washington, April 22, 1980.
7. D.L. Olson, "Pyrometallurgical Research in Welding Consumables", presented at DOE-BES Berkeley Meeting, September, 1980.

8. D.K. Matlock, "Dissimilar Metal Welding Research", presented at DOE-BES Berkeley Meeting, September, 1980.
9. D.K. Matlock, "Experimental Design for Stress Studies in Lithium", presented at Fifth Lithium Users Workshop, Westinghouse R&D Center, Pittsburgh Pa., October, 1980.

6.4 Summary of Abstracts.

1. C.E. Cross and D.L. Olson, "Modification of Eutectic Fusion Zone Microstructure", submitted for presentation at 62nd Annual AWS Meeting, Cleveland, Ohio, April, 1980.
2. J.W. Elmer, D.L. Olson, and D.K. Matlock, "Thermal Expansion Characteristics of Austenitic Stainless Steel Filler Metals", submitted for presentation at 62nd Annual AWS Meeting, Cleveland, Ohio, April, 1980.
3. C.E. Cross, D.L. Olson, and D.K. Matlock, "Fusion Zone Microstructure and Mechanical Properties of Dissimilar Metal Weldments", submitted for presentation at 62nd Annual Meeting, Cleveland, Ohio, April, 1980.
4. G.M. Campbell, D.K. Matlock, and D.L. Olson, "The HAZ of 2 1/4Cr-1Mo Steel From an In-Service Dissimilar Metal Weld", Submitted for presentation at 62nd Annual AWS Meeting, Cleveland, Ohio, April, 1981.
5. D.L. Hammon and D.K. Matlock, "The Effects of Lithium on Fatigue Crack Propagation in Iron Base Alloys", submitted for presentation at CORROSION / 81, Toronto, Canada, April, 1981.
6. P. Steinmeyer, D. Williamson, and D.L. Olson, "Corrosion Kinetics of 304L stainless Steel in Liquid Lithium as Observed by Mossbauer Spectroscopy", submitted for presentation at CORROSION/81, Toronto, Canada, April, 1981.
7. T.L. Anderson and G.R. Edwards, "Corrosion of Ferritic Steels in a Lead-Lithium Liquid", submitted for presentation at CORROSION/81, Toronto, Canada, April, 1981.

6.5 Research Assistants Graduated

D.L. Hammon, M.S. Metallurgical Engineering
T.L. Anderson, M.S. Metallurgical Engineering