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LITHIUM COMPATIBILITY RESEARCH - STATUS AND  
REQUIREMENTS FOR FERROUS MATERIALS\*

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ABSTRACT

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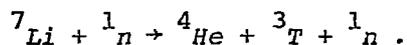
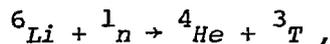
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A brief historical review and a description of the present status of the knowledge accumulated on lithium-ferrous alloy corrosion research are presented and discussed. The effects of various parameters are discussed and the future requirements of lithium corrosion research are presented and discussed.

**MASTER**

INTRODUCTION

The potential importance of liquid lithium metal in modern technology has been increasing in recent years, primarily because it is under serious consideration for both the coolant and blanket material for fusion reactor materials. This results from its neutronic properties and the reactions:



Tritium produced by these reactions is a primary constituent (along with deuterium) of the fuel in a fusion reactor and it is this breeding potential that makes the fusion reactor so attractive. The lithium atom is essential for the breeding of tritium and although various designs have been proposed in which  $\text{Li}_2\text{O}$  (1), Li-Al alloy (2), or  $\text{Li}_2\text{BeF}_4$  (3) are considered, pure lithium gives the greatest neutron efficiency and is generally favored as the blanket material (4-8). While the corrosiveness of lithium may limit its usefulness as a coolant, its breeding potential looms very important.

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At the present time it appears that first generation fusion reactors of the tokamak design will probably be constructed of 316 stainless steel or a modification of this alloy. In view of the fabrication technology developed for austenitic stainless steels compared to other potential materials, these steels will probably be specified for other fusion reactor designs, should they become feasible. In view of this, a knowledge of the compatibility of liquid lithium with ferrous alloys is necessary in the near future to avoid unpleasant surprises, when fusion reactors become a reality.

It is the purpose of this paper to attempt to summarize the status of lithium research: where we have been, where we are, and where we are going.

## BACKGROUND AND REQUIREMENTS

### Brief History of Lithium Corrosion Research

Lithium corrosion research in the U.S. began in the early 1950's because its physical properties offered unique advantages as a heat transfer medium. Its high heat capacity, low viscosity, high thermal conductivity and low vapor pressure made it a favorable candidate as a heat transfer medium for nuclear reactors, including the Aircraft Nuclear Propulsion (ANP) system.

Early work was generally of the nature of static screening tests which were typical of the period. These workers did not always appreciate the need for careful handling of lithium to minimize contamination from air or other external sources. As a result, data are often contradictory and difficult to evaluate. Nevertheless, certain fundamentals were recognized:

1. Iron and iron-based alloys could be used to contain lithium for short durations to temperatures above 300°C (9-12).
2. The presence of carbides in low alloy steels could be a source of intergranular corrosion and penetration (9,13).
3. Dissimilar metals would transfer when immersed together in lithium (14-16).

4. Higher nickel stainless steels were less resistant to general corrosion (14-16).
5. Radiation did not appear to influence the corrosion of iron (17).

The use of thermal convection loops, to test lithium under flowing conditions (18), produced the added complication of thermal gradient mass transfer. Metal dissolved in the hot portion of the loop would deposit in the cold portion of the loop and eventually plug the loop with a metallic deposit, even with a temperature gradient as low as 10°C. Intergranular attack in the vicinity of sigma phase particles and carbide precipitates was also confirmed. Intergranular attack was found to be greatly accelerated by stress in the steel.

By the mid fifties it became clear that lithium was much more corrosive toward stainless steels than sodium (19). By this time, it became possible to classify the different corrosion mechanisms operating in liquid metal systems and identify those variables that are important in lithium-ferrous alloy systems (20).

The types of liquid metal attack identified are:

1. Simple solution,
2. Alloying between liquid metal and solid metal,
3. Intergranular penetration,
4. Impurity reactions,
5. Temperature gradient mass transfer, and
6. Concentration gradient mass transfer or dissimilar-metal mass transfer.

Variables that affect liquid metal corrosion are:

1. Temperature,
2. Temperature gradient,
3. Cyclic temperature fluctuation,
4. Surface-area-to-volume ratio,

5. Purity of liquid metal,
6. Flow velocity or Reynolds number,
7. Surface condition of container material,
8. Number of materials in contact with the same liquid metal, and
9. Condition of the container material (thermomechanical treatment).

Although the impurity content of the lithium was recognized as an important variable in corrosion studies, it was not until the work of McKee (21) that quantitative data on the effect of nitrogen was obtained. Increased nitrogen resulted in increased mass transport in a type 316 stainless steel loop. In addition, the steel was found to contribute nitrogen to the lithium during the test. Above 800°C, lithium dissolves nitrogen from the steel which, in turn, increases the nitrogen content of the lithium and increases the corrosion of the steel. Thus, it is possible for a steel to serve as an agent for its own destruction.

Forced convection loops of 316 stainless steel were studied by Gill, et al. (22) who then attempted to explain their results in terms of mass transport and flow rate correlations. They concluded that mass transfer from the solid to the liquid stream is controlled by solid phase diffusion and cannot be predicted from correlations based on liquid phase diffusion. Since the precipitation process is rapid relative to the solution process, mass transfer is limited only by the concentration or activity potential available.

From about 1962 until fairly recently, most of the published work on lithium corrosion has come from Russia. In order to simplify their mechanistic studies, they eliminated temperature gradient mass transfer by the use of isothermal tests. Most of these works fall into three categories:

1. Understanding the basic mechanisms of corrosion by lithium,
2. Determining the exact effect of lithium on the properties of candidate materials, and
3. Finding means of preventing or inhibiting corrosion by lithium.

Several years ago work began again in this country on the corrosion of lithium on ferrous alloys, especially austenitic stainless steels. This work is in support of the fusion energy programs and has been carried out at the Colorado School of Mines (CSM) and the Oak Ridge National Laboratory (ORNL). The work at CSM has centered about mechanistic and kinetic studies of ferrous alloys with lithium containing high nitrogen (>2000 ppm) and the work at ORNL has been concerned with kinetic studies utilizing low nitrogen-bearing lithium (<200 ppm). Significant differences in the corrosion rate between these two works have been related to the differences in corrosive environments.

#### Information Required

Successful operation of a large engineering system such as a fusion reactor requires accurate information on the potential performance of all its components. Currently, a number of design studies are being conducted throughout the world which incorporate a number of coolants, breeder materials and container materials.

In order to properly evaluate the various combinations, specific information is required on the corrosion rates of the coolants and breeder materials on their containers. Specifically, corrosion rates under both static and dynamic conditions are required. Besides information on corrosion rates, information is required on the following:

1. The exact mechanisms of corrosion under various conditions,
2. The effect of variables on corrosion rate,
  - a. Temperature,
  - b. Time,
  - c. Interstitial impurity concentration,
  - d. Flow rate,
  - e. Temperature differential,

- f. Effects of dissimilar materials in the system,
- g. Effects of strong magnetic fields, and
- h. Mechanical stresses.

Knowledge of the effects of these variables is required in addition to the basic rate equations in order to include proper "safety factors" in the design. Assignment of proper safety factors involves not only the effects of these variables on corrosion rate, but also the identification of any synergistic effects which may occur. Ultimately, this will require detailed engineering tests designed to provide control of as many of the variables as possible.

Proper selection of materials for any proposed system must be made early in the design program. Undesirable materials will have to be eliminated early so that attention can be focused on the remaining candidates. Other selection criteria must include availability fabricability, cost and ferromagnetism.

Austenitic stainless steel, notably type 316, is the alloy most seriously considered for magnetic fusion reactors (tokomaks) at the present time. This selection is based partially on the selection criteria discussed above as well as the fact that, within certain restraints, its corrosion resistance and radiation damage resistance are acceptable. In addition, extensive use of these alloys in industry, including fission reactors, has established a broad technological base with which to operate. Several of these alloys have been approved by the ASME Code for nuclear pressure vessel applications.

In oxidizing atmospheres austenitic stainless steels have good resistance to degradation because of passive oxide layers which build up on the surface providing a protective layer. In lithium, however, passive layers are not possible because of the strong reducing capability of lithium. Therefore, corrosion resistance must be based on other factors.

In magnetic fusion reactors the high magnetic field precludes the use of ferromagnetic materials of construction which dictates that austenitic grades are essential. However, for other types of reactors where ferromagnetism does not present problems, materials such as 2 1/4 Cr-1 Mo steel have potential. For example, thermal convection loops constructed of this alloy (23) gave a maximum corrosion rate of 2.5  $\mu\text{m}/\text{yr}$  at 600°C compared to 12.7  $\mu\text{m}/\text{yr}$  for austenitic stainless steels. Use of this alloy would also require less chromium as an alloying addition, which may be of strategic concern in the longer term.

#### EVALUATION OF PRESENT STATUS

Identification of the requirements for large scale use of lithium-ferrous alloy systems provides a meaningful background against which to review the state-of-the-art of lithium corrosion. The purpose of this section, therefore, will be to delve into the various corrosion mechanisms and the effects of variables on these mechanisms. The corrosion of ferrous container materials takes on a number of forms depending on the specific environment (24). Two mechanisms commonly observed are solution or general attack and grain boundary or intergranular attack.

#### Solution Attack

In a loop the thermal gradients promote dissolution of the container wall in the hotter regions due to higher solubility and deposition from the supersaturated liquid lithium as it passes through the colder regions. Figure 1 illustrates these dissolution-mass transport-deposition processes which cause loss of material at the hot leg and possible constriction to fluid flow (plugging) at the cold leg. The actual mass transport processes are more complex for alloys for they are influenced by chemical gradients as well as thermal gradients. Many liquid lithium test loop experiments have

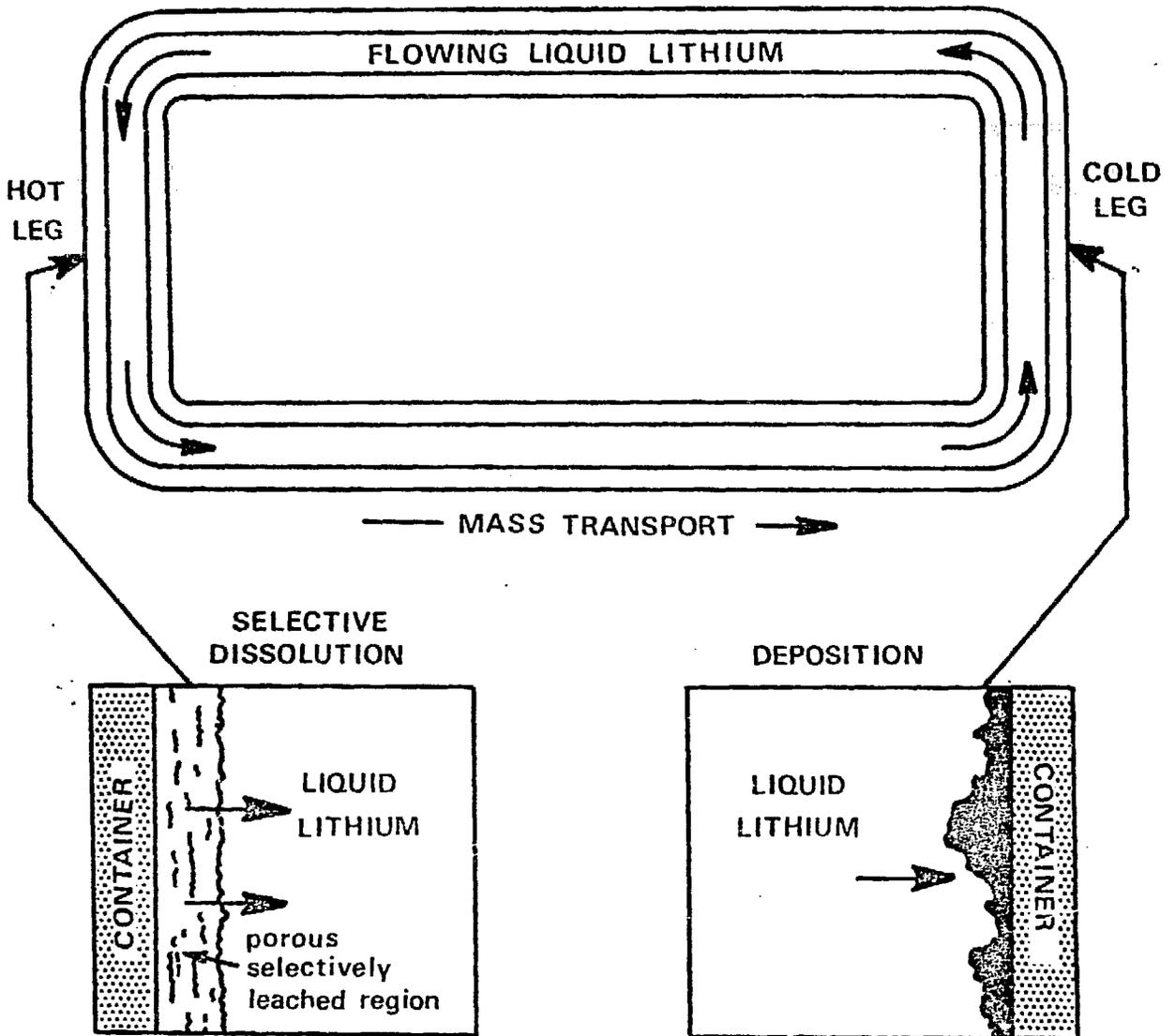


Fig. 1. Dissolution and Deposition Processes in a Thermal Convection Loop.

terminated due to plugging of the cold leg. The rate at which this corrosion process proceeds is a function of temperature, lithium purity, fluid flow-rate and alloy composition.

The rate of solution attack is very sensitive to ferrous alloy composition. Alloying elements that have large differences in solubility over the temperature range of the system are generally more subject to mass transport in lithium. Nickel and cobalt are examples of such elements and as a result superalloys generally perform poorly in liquid lithium especially at temperatures above about 500°C. In general, with increasing nickel content in ferrous alloys the susceptibility to dissolution attack increases. Other elements, even those with smaller solubilities can exhibit higher rates of dissolution attack if these elements react with impurities in the liquid lithium to produce compounds. Compound formation serves to remove the dissolved species from the liquid lithium and thus promotes accelerated dissolution of that element from the container material. Chromium from ferrous alloys is susceptible to compound formation which has been reported to enhance the solution attack when sufficient nitrogen and carbon are present in the liquid lithium. It has been demonstrated that with good interstitial impurity control of the liquid lithium, especially nitrogen, certain ferrous alloys can give excellent long-time-service as a container material.

The nature of solution attack suggests that the use of alloys with limited chromium and nickel contents is more desirable from a corrosion standpoint. In fact, pure iron has demonstrated excellent corrosion resistance in liquid lithium; but since pure iron does not have the mechanical properties suitable for pressure vessel and loop design, it is essential to search for an optimum alloy content. Figure 2 illustrates the Fe-Cr-Ni system upon which most corrosion alloys are based and indicates that our selection most likely will be centered around the iron-rich portion of the diagram. With

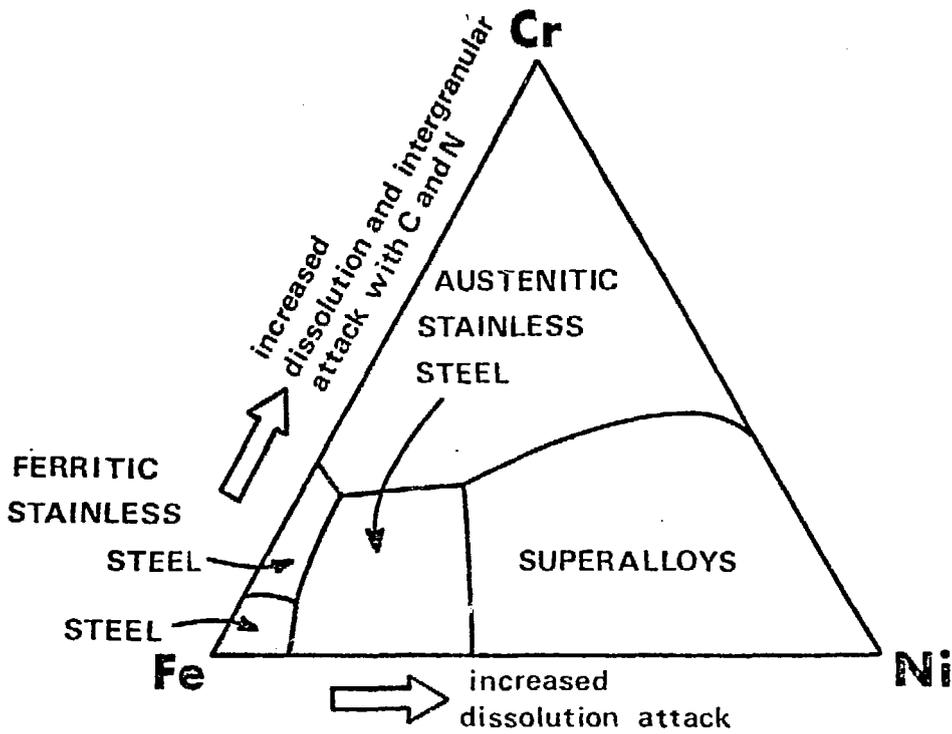


Fig. 2. Schematic Showing Relationship Between Composition, Alloy Classification and Corrosion in Lithium for Fe-Cr-Ni System.

proper impurity control in both the ferrous container alloy and in the liquid lithium, acceptable engineering alloys such as 304L and 316 stainless steel and 2 1/4 Cr-1 Mo steel have demonstrated acceptable corrosion rates in laboratory loop tests and suggest good long-term service at temperatures up to at least 500°C (23). Austenitic stainless steels are nonmagnetic and may well be required for magnetic fusion concepts. Corrosion data for these alloys are presently quite limited, and further research is necessary to understand the influence of higher fluid flow rates, structural stress and radiation damage in order to achieve dependable engineering design data.

In designing for the containment of liquid lithium, it is important to avoid the use of dissimilar alloys in the system. The chemical potential gradients of specific alloying components may become extremely large across dissimilar alloy joints and promote mass transport of these alloying constituents through the liquid lithium causing solution attack of one alloy and deposition of the alloying constituent on the other alloy. This is shown in Fig. 3. Interstitial elements are especially susceptible to this type of mass transport due to their large diffusion coefficients in both the solid container material and in the liquid lithium.

Solution and deposition kinetics have been measured in terms of weight change per unit time. Weight gains or losses may occur depending on the nature of the corrosion test; that is, whether the lithium under test is static, agitated or pumped through the thermal gradient of a loop. Careful analysis of weight change data is required since there apparently are several types of chemical dissolution and deposition mechanisms which may be involved. Micrographs of corroded surfaces indicate that the weight loss mechanisms for static and flowing lithium are different.

Schlager, et al. (25) made weight loss measurements on stainless steel in static lithium with high nitrogen concentrations in order to obtain a

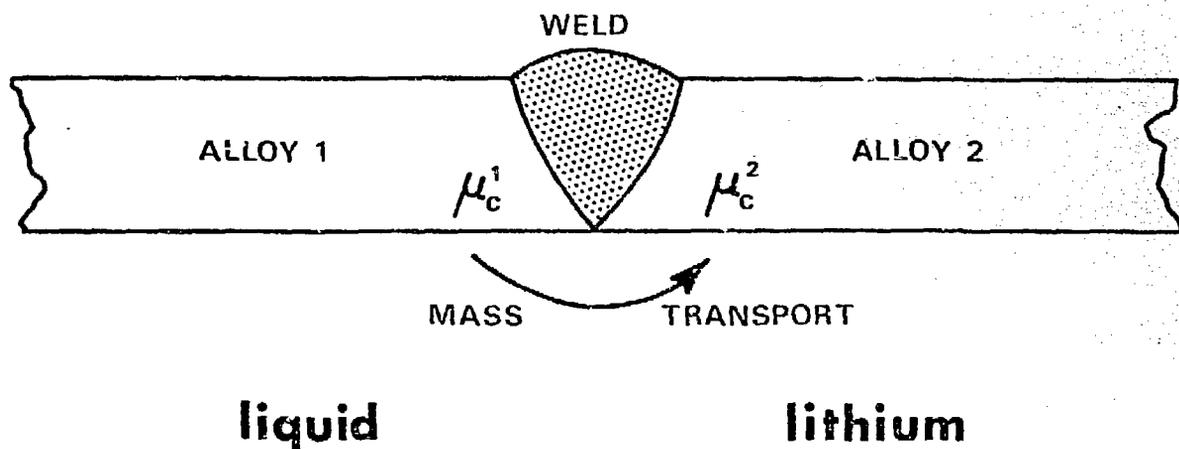


Fig. 3. Schematic Showing Dissimilar Metal Mass Transfer.

general weight loss equation. The nitrogen concentration levels used were much higher than would be used in larger systems where impurities could be monitored and controlled. Variations in nitrogen concentration were achieved by using various gettering procedures on the liquid lithium.

Liquid lithium corrosion results are often reported in mg/cm (1000 hr), which is only useful in long-time corrosion predictions if the corrosion kinetics have a linear time dependence. Therefore, it is essential to establish the correct time dependence. According to Schlager et al. (25) 304L stainless steel gives weight loss rate equations with a parabolic time dependence for time periods up to 500 hours. The weight loss (W.L.) expression for temperatures below 815°C is given as:

$$W.L. = \left( 82.2 [N]^{2.5} e^{\frac{-71 \text{ KJ/mole}}{RT}} t \right)^{1/2},$$

where  $[N]$  is the weight percent of nitrogen,  $t$  is the time in seconds,  $T$  is the temperature in Kelvins and  $R$  is the gas constant. From this equation it is apparent that the nitrogen concentration of the liquid lithium has a strong influence on the corrosion or weight loss rate but does not influence the rate controlling step or activation energy.

The above weight loss relationship is based on relatively short term corrosion tests (less than 500 hr) and relatively high nitrogen contents (500-2000 ppm) so there has been some concern as to its validity at longer times and lower nitrogen contents. However, recent tests (26) to 10,000 hr with lithium containing less than 100 ppm nitrogen were found to agree well with this relationship.

#### Intergranular Attack

Grain boundary penetration of ferrous alloys by lithium is a serious concern when the liquid lithium contains over 500 ppm nitrogen (27,28). The

grain boundary penetration at higher nitrogen concentration proceeds well ahead of the general or dissolution attack into the material as illustrated in Fig. 4. It is obvious from bend specimens that there is no mechanical integrity along the penetrated grain boundaries.

Anderson and Stephen (9) were the first to observe a relationship between grain boundary penetration by lithium and the sensitization behavior by stainless steel. They found increased grain boundary attack in the temperature range of sensitization. Other supporting evidence that intergranular penetration and corrosion are related to the sensitization of the stainless steel is found in the fact that the special grades of stainless steels such as types 304L, 321 and 347 (37,24) exhibit superior corrosion resistance to grain boundary attack.

The role of sensitization in the intergranular corrosion of stainless steel in lithium has been explained by two different models. The first model (29) suggests that sensitization leaves the region adjacent to the grain boundaries deficient in chromium and higher in effective nickel which in turn is selectively leached out by the dissolution process, leaving the regions along the grain boundaries with a semi-porous corroded ferritic structure with little mechanical integrity. This mechanism is illustrated in Fig. 5. The other intergranular corrosion model (7) is based on the concept that lithium reacts with grain boundary carbides to form less dense  $\text{Li}_2\text{C}_2$  grain boundary corrosion product which is susceptible to cracking as illustrated in Fig. 6. This suggests that grain boundary penetration follows grain boundary carbide formation which is consistent with the penetration kinetics of 304L stainless steel results of Patterson et al. (27) which indicate that there is a delay time prior to grain boundary penetration as shown in Fig. 7. Figure 8 shows the C-shaped nucleation type curve for penetration delay time as a function of temperature which is compared with the nucleation curve for carbides in stainless steel (9).

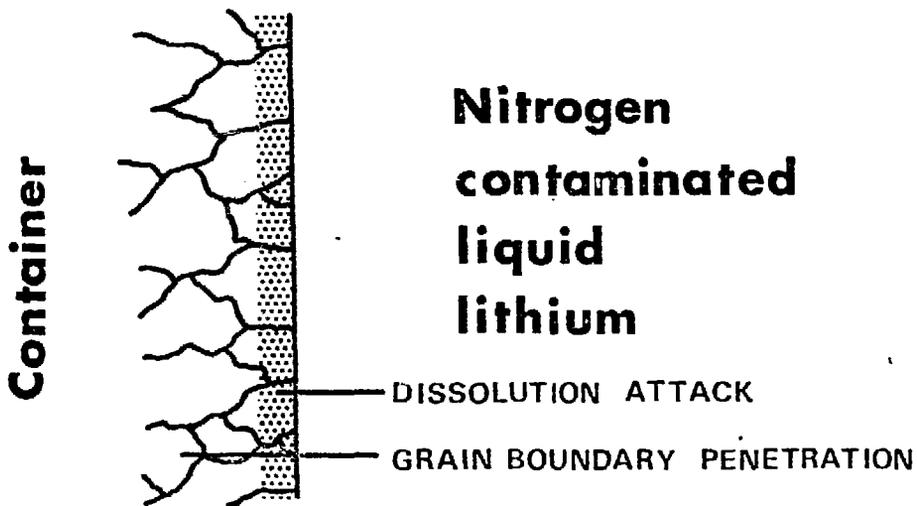


Fig. 4. Relationship Between Grain Boundary Penetration and Dissolution Attack.

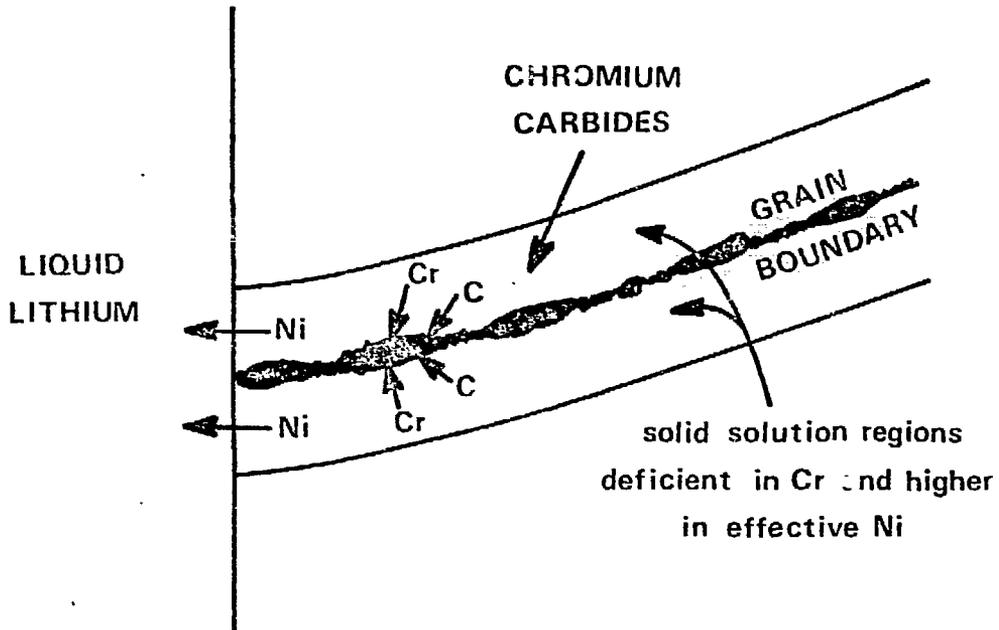


Fig. 5. Selective Leaching of Nickel Rich Areas Surrounding Chromium Carbide Phases.

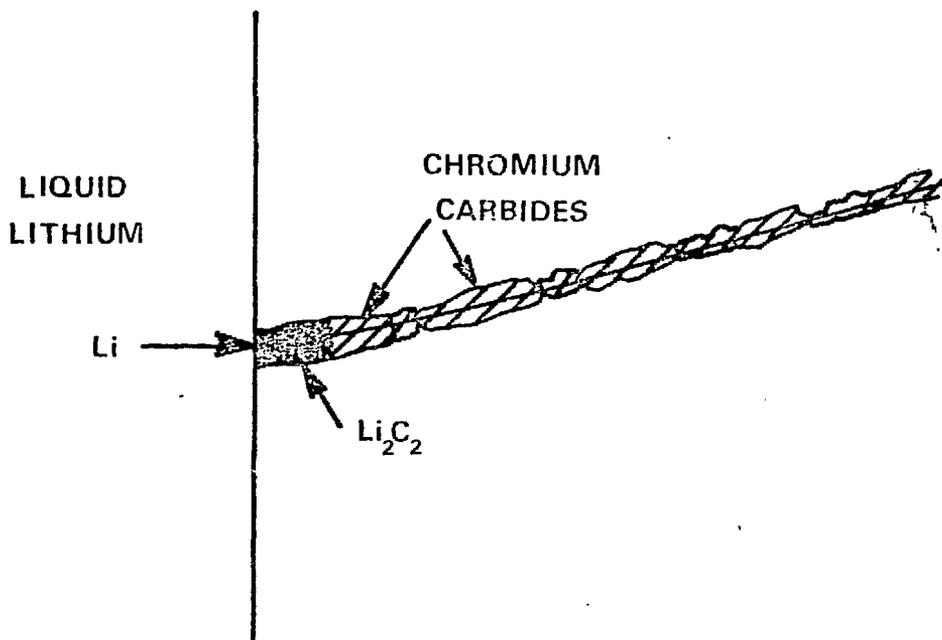


Fig. 6. Lithium Reaction With Chromium Carbides.

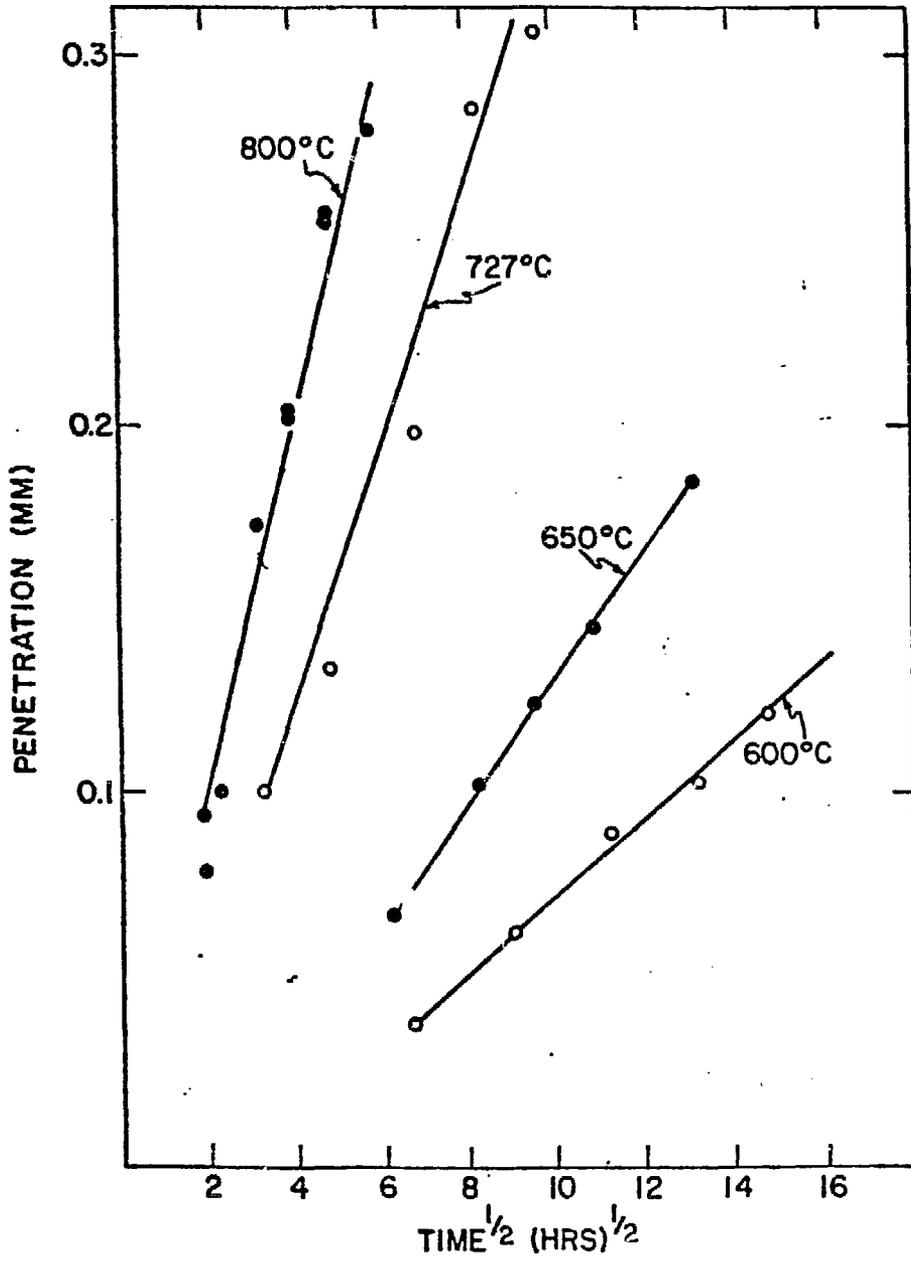


Fig. 7. Grain Boundary Kinetics for 304L Stainless Steel. Extrapolation of the lines to zero penetration suggests a delay time for penetration.

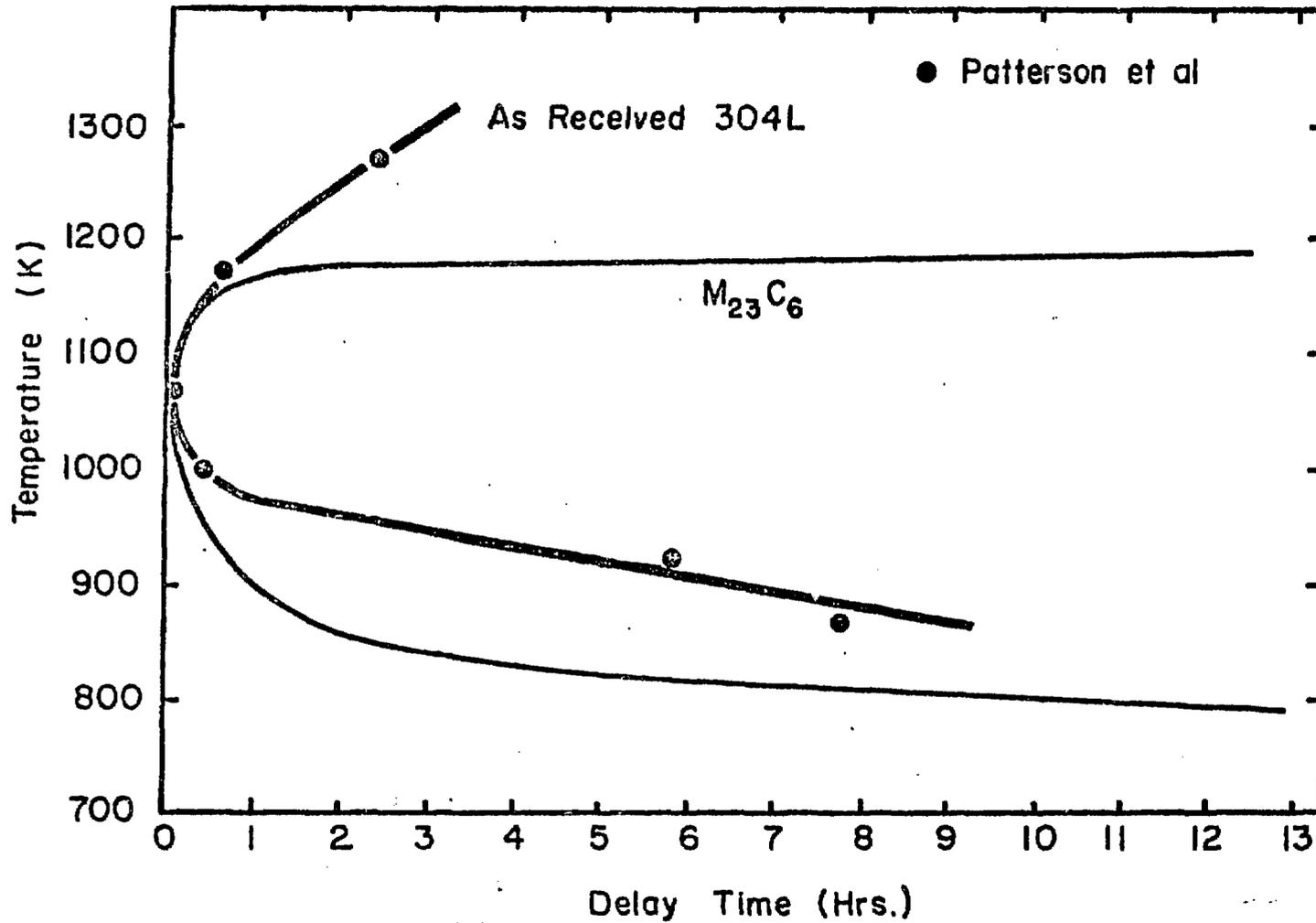


Fig. 8. The Delay Time as a Function of Temperature for the Grain Boundary Penetration in As-Received 304L Stainless Steel is Compared to the Time to Nucleate a Metal Carbide in 316L Stainless steel.

Since chromium nitrides may also be at the grain boundaries, intergranular attack can also be explained by lithium reaction with chromium nitride to form complex nitrides. Hoffman (24) has suggested that the corrosion products such as  $\text{Li}_3\text{N}\cdot\text{FeN}$  may be involved in lithium corrosion. The source of carbon or nitrogen may be either the stainless steel or the lithium.

A better understanding of the role of carbon is essential since it would be an advantage to use normal grades of stainless steels in which carbon is present as a high temperature strengthener. Since sensitization of stainless steel is an interim effect, the chromium-depleted regions are replenished at long times and elevated temperatures by transport of chromium from the interior of the grains. If corrosive attack is the result of chromium depletion and nickel leaching (Fig. 5), thermal stabilization prior to lithium service may provide a solution. However, if corrosion occurs by lithium attack of carbides as shown in Fig. 6, then a more expensive stainless steel with stabilizing additions and lower carbon content must be considered.

#### Effects of Stress

The mechanical behavior of ferrous alloys has been reported to be altered by liquid lithium exposure (30,31,32). Also, the corrosion rates of ferrous alloy specimens under stress have been reported to be greater than for unstressed specimens (33). Pure iron specimens which show excellent corrosion resistance, as measured by both weight loss and grain boundary attack, were stressed by various methods of loading and show increased grain boundary penetration rates (32,34). This is shown in Fig. 9. This corrosion rate increase has been related to the creep behavior of iron. In fact, the grain boundary penetration rate coefficient was found to be

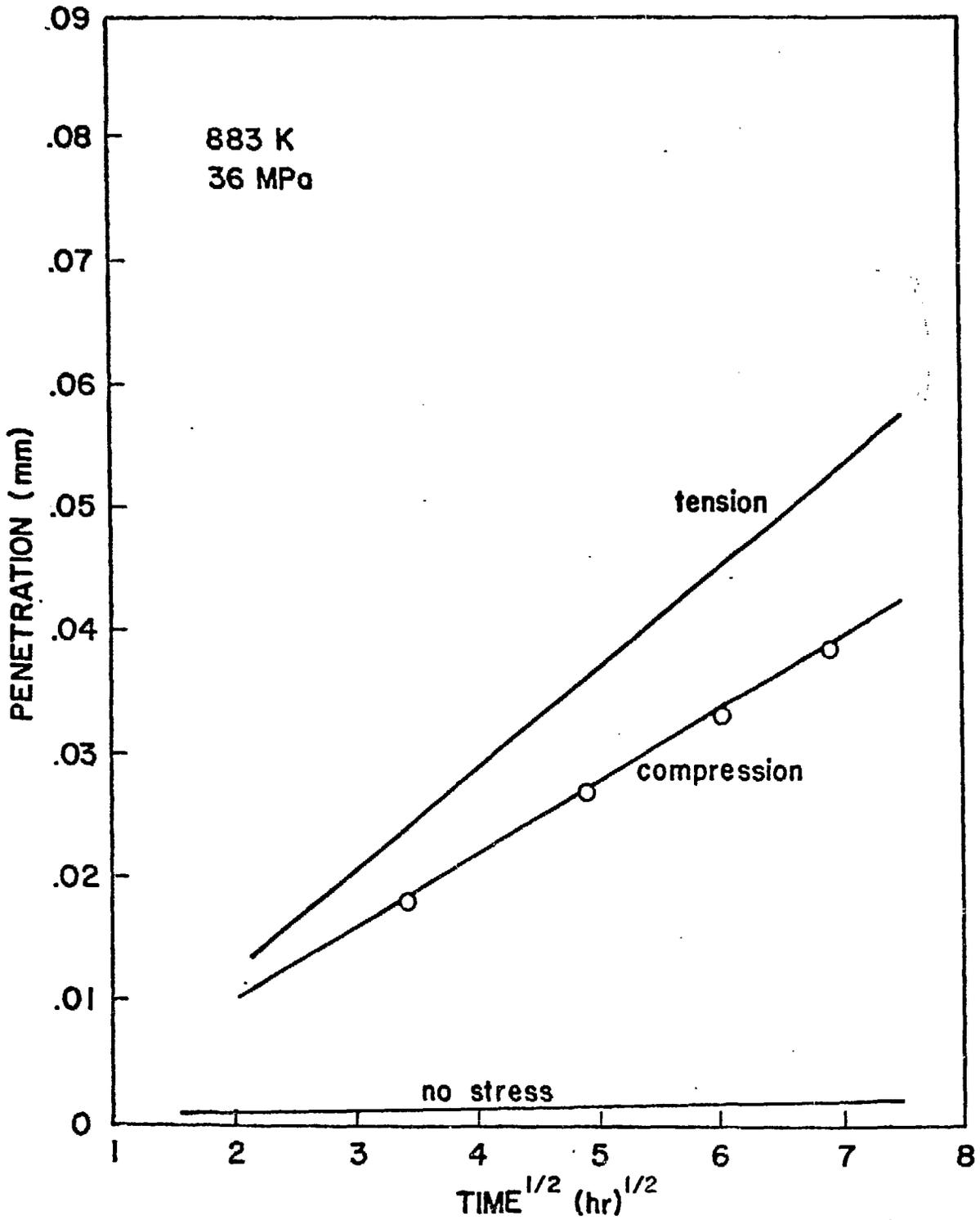


Fig. 9. Effect of Loading Method on Grain Boundary Penetration in Nitrogen Contaminated Lithium.

directly related to the strain rate as shown in Fig. 10. The major concern with these results is that the stress level at which this stress-induced corrosion process occurs is within the normal residual stress levels for this material. This raises serious questions as to the ability of the present unstressed liquid lithium corrosion data to predict the service life of lithium container materials. The corrosion behavior associated with weldments subject to constraints especially needs to be better established. It is important to understand that this stress enhanced corrosion behavior was observed only during the creep of pure iron. There are no stress related corrosion data at the present time for either low alloy or stainless steels.

#### Role of Interstitials

The effects of the interstitials nitrogen and carbon on lithium corrosion have already been discussed in detail. However, the effects of oxygen and hydrogen are less clear. Goickman (31) found penetration of lithium into 321 stainless steel to be accelerated by oxygen, although not as badly as by nitrogen. When both oxygen and nitrogen were present the penetration increased even more. Recent static tests at ORNL showed no apparent effect of 0.09 wt % oxygen on the weight loss of type 316 stainless steel specimens. Similarly, hydrogen produced no adverse effect on 316 stainless steel.

#### Fluid Flow

Several investigators have reported that flowing lithium is more corrosive than static lithium (31,33), but no definitive study has been made on the quantitative effect of flow rate on either the corrosion rate or corrosion mechanisms. This is one area requiring further work in order to provide design information.

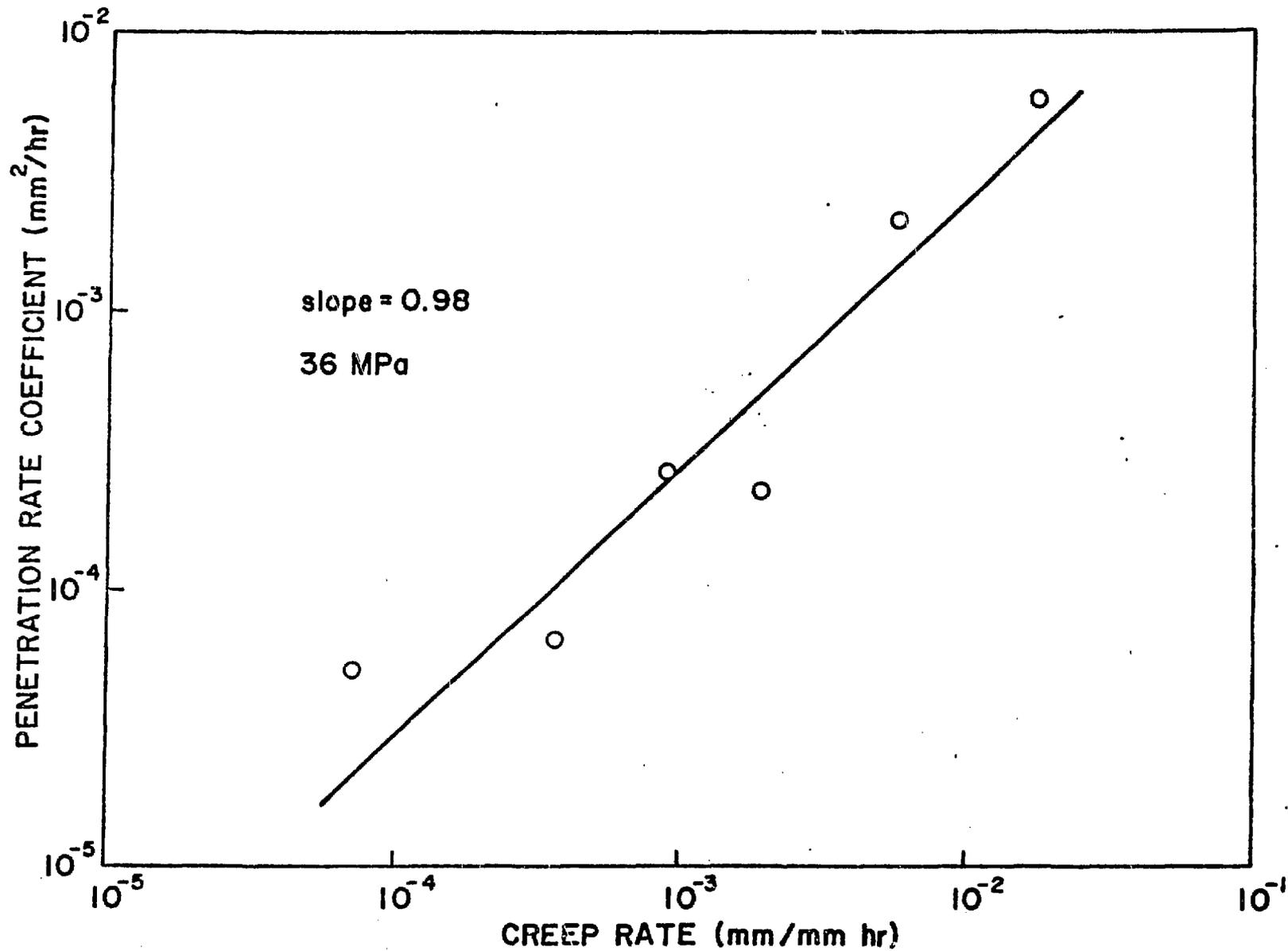


Fig. 10. Relationship Between Penetration Rate Coefficient and Creep Rate.

The effect of a strong magnetic field will be to reduce turbulent flow to a minimum and to reduce the shear layer at the liquid-solid interface. No work has been done to examine these magnetic field effects on corrosion kinetics.

#### Radiation Damage

Several investigators have indicated that irradiation produces no effect on the corrosion of steels by lithium (35,36). However, these tests were conducted at relatively low thermal neutron fluxes. Since fusion reactor first wall materials will be subjected to 14.1 MeV neutrons at much higher flux levels, possible adverse effects on corrosion should be investigated.

#### Dissimilar Metals

While it is generally desirable to avoid the presence of dissimilar metals within the same liquid metal system, it is sometimes unavoidable for a variety of reasons and sometimes may even be desirable. As already discussed, dissimilar metals present in the same system can result in transport from one part of the system to another because of differences in chemical potential.

It is possible that the use of getters in the system may prove to be necessary in order to maintain adequate purity levels in the lithium or to remove tritium generated within a fusion reactor blanket. Mass transport can occur to either plug the loop or contaminate the surface of the getter and destroy its effectiveness. Gettering materials are usually solids, but liquid getters are not beyond the realm of possibility. Recent work at Oak Ridge (26) has shown that the addition of 9.25% calcium, which is soluble in lithium, can reduce the adverse effects of 2% nitrogen considerably. Further work on these systems is necessary.

The addition of 4.8% aluminum, which is also soluble in lithium, to lithium bearing static capsules has resulted in mass transport of aluminum to the 316 stainless steel specimens to form an aluminum bearing intermetallic compound which has the potential for protecting the steel from further attack. Addition of aluminum to a thermal convection loop, operating with a hot leg temperature of 600°C with a 200°C  $\Delta T$ , resulted in a reduction in the weight lost from hot leg specimens by a factor of 4. Thus, this approach shows some potential for reducing the corrosion rate. However, considerable work must be done to establish limitations to the use of this technique and to optimize the conditions. Other elements may also prove to be useful, but these can only be established by experiment.

#### Alternate Approaches

Another method of describing the rate of weight change at various locations in a loop is through the use of mass transfer rate equations. Using the equation of continuity and transport rate expressions, empirical rate coefficients can be determined. This procedure may be valuable to assist in predicting liquid lithium corrosion behavior for fusion reactor scale-up. This approach may also be useful to extrapolate corrosion rates under various lithium flow conditions, but will not produce mechanistic understanding of the rate controlling process. It would be extremely valuable to couple such rate phenomena experiments with carefully designed mechanistic (activation energy) investigations to achieve both an understanding of the role of specific interstitial elements as well as to identify limitations of various design criteria.

#### SUMMARY

We have attempted to give a brief review of the status of lithium research; how we got to where we are and where we need to go in the future.

We are now at the point of understanding what parameters affect the corrosion of ferrous alloys by lithium and are just beginning to understand the effects of these parameters on the mechanisms and rates of corrosion.

Future needs of the fusion reactor program require the following:

1. Identification of corrosion mechanisms,
2. Establish kinetics of various reactions,
3. Determine effects of different variables,
4. Determine proper scaling factors,
5. Proof testing under reasonably accurate operating conditions, and
6. Investigate alternate methods of minimizing corrosion.

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