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A TECHNIQUE FOR CORROSION TESTING IN LIQUID LEAD

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A TECHNIQUE FOR CORROSION TESTING IN LIQUID LEAD

J. V. Cathcart W. D. Manly

SUMMARY

A new testing technique has been devised which was found to be satisfactory for comparing the dynamic corrosion characteristics of a series of metals in liquid lead. The tests were conducted in small quartz thermal-convection loops. The necessary apparatus was simple, easy to construct, and relatively inexpensive. In addition, the method afforded a means of studying metals such as chromium and molybdenum which, because of difficulties in fabrication or lack of oxidation resistance, could not be tested by more conventional techniques.

It has long been evident that the thermal properties of liquid metals should make them excellent as heat-transfer media. Work in this field was pioneered by the development of the mercury boiler.[1] In recent years the use of liquid metals as coolants in nuclear reactors, as well as in a variety of industrial applications, has received serious consideration.[2]

The increasing interest in liquid metals has focused attention on the accompanying corrosion problems and the techniques for corrosion testing in these materials. One of the most serious types of corrosion encountered has been that of mass transfer, that is, the transport of container material from the hot to the cold zone of the system under consideration. Any testing program designed to determine the most suitable container material to be used with a given liquid metal must take mass transfer into account; therefore the testing apparatus must provide for the circulation of the liquid metal through a temperature gradient. Simplicity of design and operation are also important, since a large number of experiments are frequently required before a choice of the proper container material can be made.

The apparatus described in this paper meets the foregoing specifications for tests involving liquid lead. It is a modification of the thermal-convection loops which were used extensively in the development of the mercury boiler.[1,3] As shown in Fig. 1, the loop was made of ½-in. quartz tubing. Six-

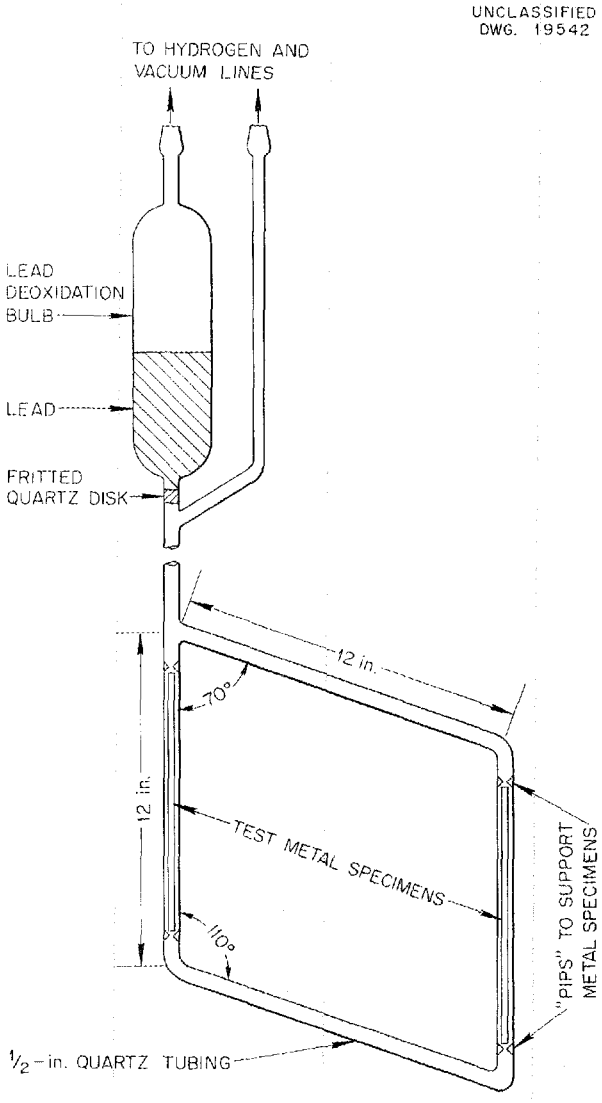

Fig. 1. Quartz Thermal Convection Loop.

[1] H. N. Hackett, *Trans. Am. Soc. Mech. Engrs.* 64, 647 (1942).

[2] R. N. Lyon (ed.), *Liquid Metals Handbook*, p 1–28, Government Printing Office, Washington, 1950.

[3] R. C. Reid, *Mech. Eng.* 73, 514 (1951).

inch tubes of the test metal were mounted in the hot and cold legs of the loop, where they were held in place by indentations in the quartz tubing. Attached above the loop and separated from it by a fritted quartz filter disk was a large quartz bulb, 80 mm in diameter. This bulb was used for the deoxidation of the lead prior to the loading of the loop. Two side arms, one above the deoxidation bulb and the other below the fritted disk, were attached to hydrogen and vacuum lines through a suitable system of stopcocks. Eight-inch split-core clamshell heaters were used to heat the loop, one such heater being suspended by stainless steel clamps around each arm of the loop. A fifth heater was placed around the tube connecting the loop and the deoxidation bulb. The bulb was heated with electric tape heaters. Temperatures were measured by thermocouples spaced around the loop and tied in place with wire. The temperature of the hot leg was controlled with a Leeds & Northrup Micromax controller operating in conjunction with a No. 10769-G control unit.

With the loop set up in operating position, the lead in the deoxidation bulb was heated to 425°C, and hydrogen was allowed to pass through the fritted quartz disk and to bubble up through the lead. During the deoxidation process, which lasted for about 48 hr, surface-tension forces prevented the liquid lead from passing through the fritted disk. When the deoxidation was complete, the loop was evacuated and hydrogen was admitted into the deoxidation bulb; this caused the lead to flow through the fritted disk and into the loop. The loop having been preheated to operation temperature, the heaters on the cold arms were turned off, and circulation of the lead was maintained by the thermal-convection currents thus induced.

The above procedure assured that the oxide content of the lead was reduced to a minimum and, in addition, prevented reoxidation of the lead during the loading of the loop. The technique also offered several other advantages. Each loop required only a small amount of metal tubing, a factor of some importance in cases where the test metal is expensive or in short supply. No bending or welding of the test metal was required; therefore such hard-to-fabricate materials as metallic chro-

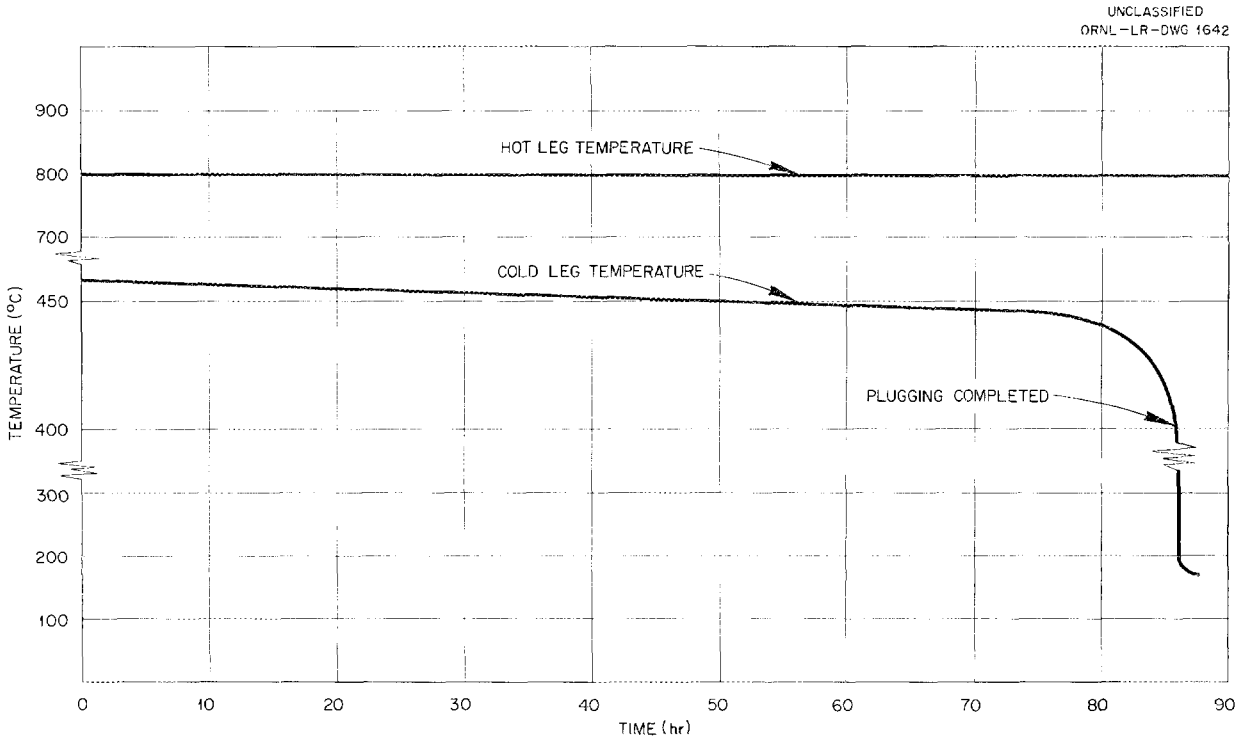

Fig. 2. Temperature Record for Lead–Type 304 Stainless Steel Loop No. 2.

mium or molybdenum could be studied without difficulty. The fact that the test specimens were completely enclosed in the quartz tubing of the loop eliminated any necessity for protecting the outer surface of the test metal from air oxidation; thus the testing of metals such as molybdenum or columbium was greatly facilitated. The detection of leaks in a quartz loop prior to loading was also much easier than in a conventional metallic loop.

The relative resistance to mass transfer of one test metal as compared with another was obtained by measuring the time required for a sufficient quantity of mass-transferred material to build up in the cold leg of the loop and cause a stoppage in the circulation of the liquid lead. This process is referred to as "plugging." The plugs were always a porous mat of fine dendritic needles and plates. A wide variety of metals and alloys has been tested by this method. A wide range of plugging times was observed; the loops containing nickel plugged in less than 2 hr, while no mass transfer at all occurred in over 600 hr in the columbium loop. Thus the technique possessed a reasonable degree of sensitivity.

The extent of mass transfer could be estimated very roughly during the course of an experiment from measurements of the cold-leg temperature. As a plug built up, the circulation of the lead was slowed, thus producing a decrease in the cold-leg temperature (see Fig. 2). Such behavior was found to be typical of plug formation. When plugging was complete, a sharp drop in the cold-leg temperature occurred, and the lead in the cold leg solidified.

After a test had been completed, the loop was cut into short sections, and each section was placed in a tube such as is shown in Fig. 3. These tubes were divided into two compartments by fritted glass filter disks, and each compartment was connected to hydrogen and vacuum lines through a side arm. With the tube filled with hydrogen, the lead in each section of the loop was melted, and by proper manipulation of hydrogen pressure and vacuum, the molten lead was then filtered into the bottom compartment. In this manner any mass-transferred material could be recovered and its position in the loop located.

The mass-transferred material in every loop which was operated was found to have collected in the bend at the bottom of the cold leg. In no case was there evidence of appreciable deposition on the cold-leg test specimen. These facts were taken to indicate that most of the material was deposited in the cold leg through nucleation in the bulk of the liquid lead rather than at the walls. Plug formation appeared to be a matter of mechanical agglomeration of material in the bend at the bottom of the cold leg.

Although a large number of metals and alloys have been tested by this method, only the results obtained with type 304 stainless steel will be presented here. The data from the other tests will be published as a separate paper.

Two loops containing type 304 stainless steel test specimens were operated. The hot- and cold-leg temperatures were 805 and 490°C, respectively, for Loop No. 1, and they were 800 and 450°C, respectively, in the case of Loop No. 2. Sufficient

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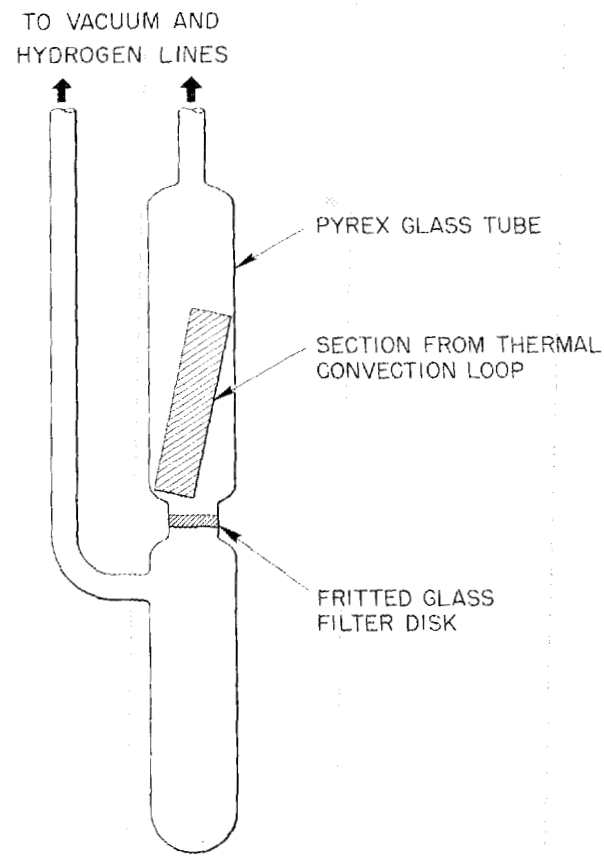

Fig. 3. Apparatus for Removing Excess Lead from Loop Sections.

Fig. 4. Transverse Section of Hot-Leg Specimen from Lead–Type 304 Stainless Steel Loop No. 2. 500X

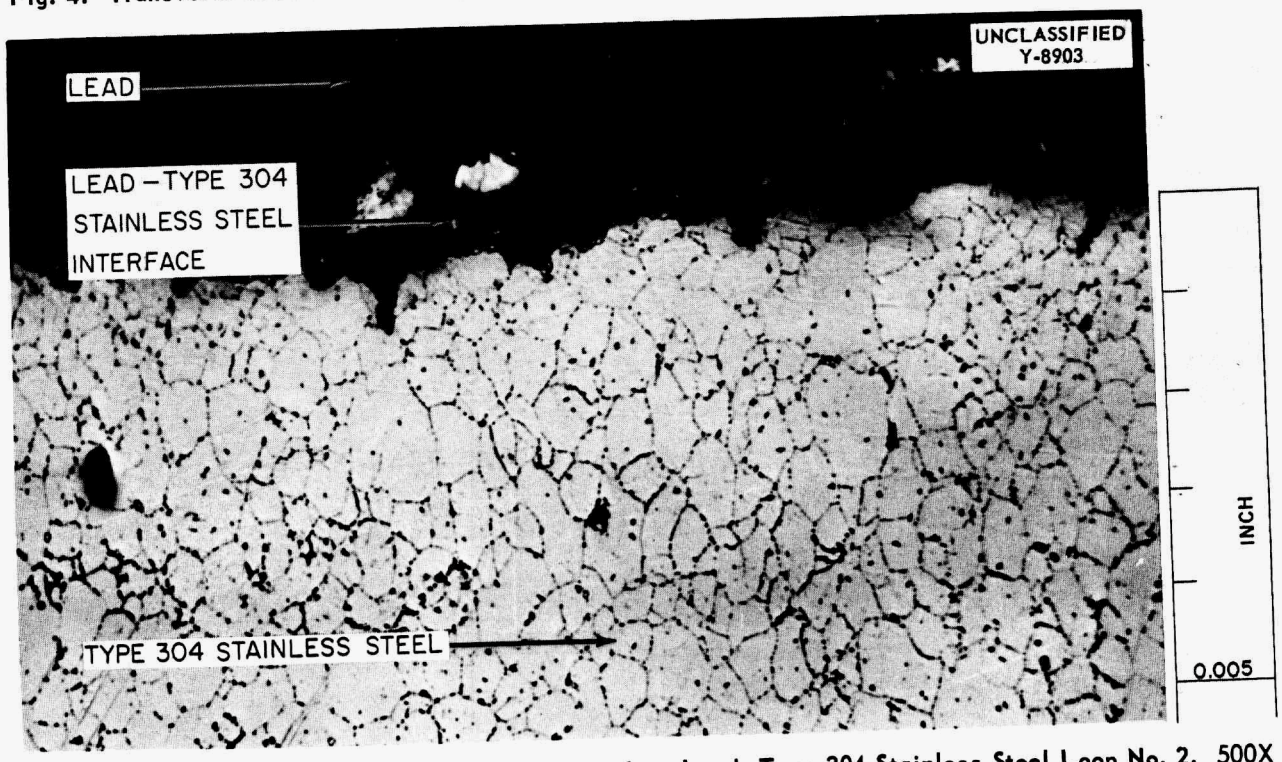

Fig. 5. Transverse Section of Cold-Leg Specimen from Lead–Type 304 Stainless Steel Loop No. 2. 500X

mass-transferred material to stop circulation of the lead collected in the cold leg of the first loop in 111 hr. The second loop plugged after 86 hr of operation. Figures 4 and 5 show transverse sections of the test specimens from the hot and cold legs, respectively, of Loop No. 2. The extensive corrosive attack of the lead is especially apparent in the hot-leg specimen. Figure 6 is a photograph of the plug taken from the cold leg of Loop No. 2. As indicated previously, this plug consisted of a mass of fine needles and platelets wedged together at the bottom of the cold leg of the loop.

CONCLUSIONS

The testing technique described in this paper has been used successfully in a comparative study of the corrosion characteristics of a series of metals and alloys. The apparatus required was simple and relatively inexpensive. The method also afforded a means of testing metals which, because of their physical properties, could not be conveniently studied by more conventional techniques.

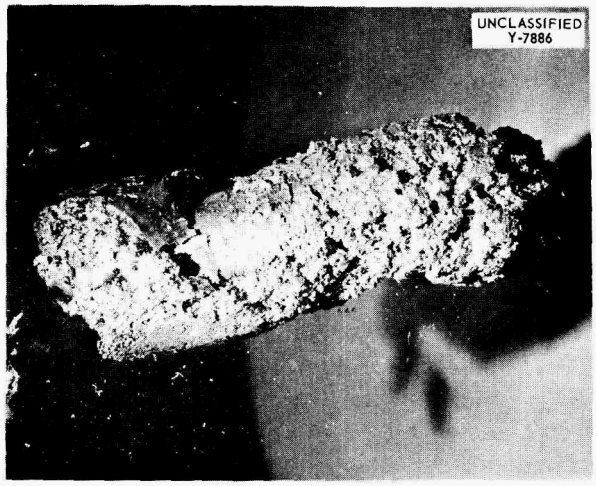

Fig. 6. Plug Taken from Cold Leg of Lead–Type 304 Stainless Steel Loop No. 2. 3X. Reduced 29%.