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MATERIAL SELECTION AND PROCESSING FOR THE
MOLTEN LAVA/SINGLE TUBE BOILER EXPERIMENT

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ABSTRACT

Materials selection for the Molten Lava/Single Tube Boiler Experiment was made on the basis of compatibility tests between Hawaiian Tholeiitic Basalt (HTB) and metal discs at 1300°C for periods up to 95 hours in air. The compatibility test showed that the HTB acted as an effective carrier of oxygen to the metal surface. High chrome alloys developed a tight Cr_2O_3 layer that was protective, so alloys for use in oxidized lava could be selected to a first approximation on the basis of their oxidation resistance in air. Based on the results of these experiments, alloy strength, and alloy fabricability, type 310SS was selected. The tube nominally was 3.00" OD, 2.50" ID, and 24" long. It was welded by Gas Tungsten Arc without filler and pressure tested to 400 psi.

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TABLE OF CONTENTS

	<u>Page</u>
Acknowledgments	2
List of Figures	5
Introduction	6
Experimental Procedures	6
Experimental Results	9
Tube Fabrication and Performance	15
References	16

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Lava Samples - Crucible	7
2	Lava-Crucible Interface	8
3	Austenitic Stainless Steel - .5 hr 1300°C	9
4	Kovar Microprobe Results - .5 hr 1300°C	10
5	Nickel Microprobe Results - 19 hr 1300°C	12
6	Fe 26% Cr Microprobe Results - 19 hr 1300°C	13
7	Nickel-Lava Interface - 1300°C	14

MATERIAL SELECTION AND PROCESSING FOR THE MOLTEN LAVA/SINGLE TUBE BOILER EXPERIMENT

INTRODUCTION

The Molten Lava/Single Tube Boiler Experiment (ML/STBE) was designed to obtain heat transfer properties of molten Hawaiian Tholeiitic Basalt (HTB). The experimental design is described elsewhere.¹ The materials selection and fabrication of the boiler tube were critical since a tube failure would have presented a significant safety problem. The expected operating conditions for the boiler tube were nominally 60 psi and 300°C but the remote possibility of temporary loss of coolant causing the tube to come to thermal equilibrium with the molten HTB indicated the need for some compatibility tests.

Initial material selection was made from the Fe, Cr, Co, Ni alloy system since the most economical materials were preferred. Thus the refractory metals and metals requiring protective coating systems were ruled out. It is important to note that the optimum material for the ML/STBE would probably not be optimum for use in deep magma because of the difference in oxygen activity. The HTB would be saturated with oxygen in the ML/STBE so the compatibility tests simulated these conditions. Other work involving material selection for the devices in magma have naturally used different experimental conditions.² The general guideline used for selecting corrosion resistant alloys follows one of two paths. One involves alloys that develop passive or protective layers as with the stainless steels. The other involves alloys that are stable or metastable toward the media. For instance, Ni or Co should be reasonable choices for use in magma since its oxygen activity is so low that NiO or CoO can not form.

EXPERIMENTAL PROCEDURES

The following metals were tested under the conditions listed:

Material	Time at 1300°C		
	1/2 hr.	19 hrs.	95 hrs.
99.5 Ni (Grade A)	X	X	X
18 Cr 8 Ni bal Fe (Austenitic)	X	X	X
26 Cr 1 Mo bal Fe (Ferritic)	X	X	X
20 Cr 80 Ni (Nichrome)			X
29 Ni 17 Co bal Fe (Kovar)	X		

The form of the metal specimen was determined in some cases by availability but the preferred specimen was a hollow cylinder, 1/2" OD, 1/4" ID, 3/8" long. The lava was cut into cubes and melted in a 99% + Al₂O₃ crucible. (Fig. 1) The metal specimen was inserted after the lava melted.

Figure 1. Lava Samples and crucible. 3X

Selection of inert crucibles for remelting lava with high iron contents can present problems.³ However, there were no difficulties with lava attacking the crucible in these experiments as long as 99+% alumina crucible was used. The lava wetted the crucible but did not penetrate it. (Fig. 2) Attempts to use crucibles made from 95% alumina were not satisfactory because of visible penetration. Under these oxidizing conditions a platinum crucible was also satisfactory but showed no advantage over the less expensive 99+% alumina crucible.

During an experiment where the temperature of the lava was measured by immersion of an unsheathed chromel alumel thermocouple, complete dissolution of the anode was observed. To confirm this as an electrochemical effect, the thermocouple was driven with a current of opposite polarity; the other lead dissolved. This phenomenon is quite reasonable when one considers the high conductivity of lava at 1300°C ($.03\text{ } \Omega^{-1}\text{cm}^{-1}$) and confirms the need to design corrosion experiments to avoid unwanted galvanic effects.⁴ A study of the electrochemical behavior of lava in a fuel cell, recently reported by Haaland,⁵ further illustrates this point.

Figure 2. Lava-Crucible Interface. 10X

EXPERIMENTAL RESULTS

At the completion of the experiment each metal specimen was examined metallographically and, in certain cases, by electron microprobe.

The experimental results will be reported by alloy and length of test. The first series of tests lasted one half hour at 1300°C. No significant attack occurred on the Ni, austenitic 18Cr-8Ni stainless steel or ferritic 25Cr-1Mo stainless steel specimens. Figure 3 shows a cross section of the austenitic stainless steel. The Kovar specimen, however, was quite interesting. The wire specimen suffered grain boundary melting but even with this disintegration microprobe analysis showed little movement of Ni or Co into the lava. (Fig. 4)

Figure 3. Austenitic Stainless Steel - 1/2 hr. at 1300°C. 80X

Figure 4. Kovar Microprobe Results - 30 min. 1300°C

3

This result was very encouraging since it had been hoped that Ni and Co would not interact strongly with the magma, particularly in the first half hour test where the HTB was not saturated with oxygen. Kovar was dropped from this test series because of the intergranular melting.

The 19 hour test showed all the alloys performing satisfactorily but revealed the different protection methods mentioned earlier. The Ni had a very low reactivity as can be seen from Figure 5. Some reaction with impurities occurred but Ni in general did not interact with the HTB. The two stainless steels developed a protective Cr_2O_3 layer which can be seen in Figure 6.

Figure 5. Nickel Microprobe Results - 19 hr. 1300°C

Figure 6. Fe-26Cr Microprobe Results - 19 hr. at 1300°C

Since there was some question as to the rate of oxygen saturation of the HTB, some 95 hour tests were performed to determine how Ni would behave. (Fig. 7) Electron microprobe studies showed Ni loss and a Ni rich phase in the HTB. It was felt then that upon saturation of the HTB with oxygen the lava acted as a carrier of oxygen to the Ni surface where it was oxidized.

Figure 7. Nickel-Lava Interface - 95 hr. 1300°C - 250X

Though the results of these tests indicated that either Ni or stainless steels had satisfactory compatibility for the ML/STBE, type 310SS was selected because of its lower cost and greater availability.

TUBE FABRICATION AND PERFORMANCE

The tube dimensions were nominally 3" OD, $2\frac{1}{2}$ " ID, by 24" long. The end closure was made by welding a $\frac{1}{2}$ " thick 310SS plug into one end of the tube. Welding was performed by the GTA process without filler to minimize chemistry variations. This practice resulted in a fusion zone with little or no delta ferrite and special care had to be taken during welding to avoid hot cracking. A 1200°F preheat and post heat was used followed by a slow cool to 600°F prior to water quenching to minimize thermal stresses. The welding was carried out with three passes using 165 amp SPDC and an argon flow of 22 CFH. After welding the tube was tested at a pressure of 400 psi.

The tube performed as expected and no discoloration occurred during use in the ML/STBE (Ref. 1). It is concluded that 310SS welded by GTA makes an adequate heat exchanger for use in a laboratory test on oxidized lava at 1300-1400°C. Additional experiments would be necessary to insure adequate performance of heat exchangers for field test on lava lakes due to the presence of acid, gases, and holoogens. These tests, as well as additional compatibility tests in low oxygen environments will be reported in the future.

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