Design and Implementation of an Experimental Electrochemical Setup for Molten Salt Corrosion Testing

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Background

Corrosion is a problem that affects multiple industries and has an estimated global cost of $2.5 trillion annually. Due to this enormous cost, it is imperative to understand the effects of corrosion on different materials. There are various methods of testing corrosion behavior, the most straightforward being immersion testing in which the material of interest is placed in corrosive media for a pre-determined amount of time. While this method is the easiest to execute, it has issues due to the inaccuracy in removal of corrosion product.

Due to the fact that corrosion is essentially a loss of electrons, electrochemical methods can also be used to quantify corrosion behavior. Electrochemical testing is a faster and more accurate method in which a potential is applied between the material of interest and a reference electrode while current is measured simultaneously. Typically, an electrochemical test can be done in as little as 1.5 hours and can yield more relevant data.

For room temperature testing, various companies sell standardized test cells which are easy to obtain and use; however, for high temperature electrochemical testing no such cell exists, thus an experimental setup for this type of experiment must be designed and built by the user. Molten salt corrosion testing requires a setup that can withstand temperatures of 600°C and above while still maintaining the parameters necessary for electrochemistry i.e. a three electrode cell, ability to attach a potentiostat, and ability to achieve the desired testing atmosphere. The design and implementation of this experimental setup is critical in observing high temperature corrosion phenomena.

Objective

To design and build an experimental electrochemical test setup which is suitable for high temperature corrosion applications.

Electrochemical Cell & Potentiostat

The cell consisted of a working, reference, and counter electrode which were all suspended in an alumina crucible. The crucible also had sufficient room to hold the corrosive media, in this case a molten salt. The working electrode is the material that is being tested. The counter and reference electrodes were wires made of platinum, which is an inert material. The reference electrode is ideally isolated from the bulk solution while still maintaining the ability to transfer electrons. In a standard electrochemical cell this can be accomplished using a thin capillary tube and a salt bridge; however, these typical reference electrode systems can be unstable at high temperatures. For the designed high temperature setup, a quartz sleeve was placed around the reference electrode to isolate it. In theory, when the salt becomes molten during testing the quartz sleeve allows for ion transfer while isolating the reference electrode from the bulk solution.

To run electrochemical tests, a potentiostat is attached to the electrodes and potential is applied between the working and reference electrodes. The current measured between the working and counter electrodes can then be used to derive a corrosion rate.

Gas Scrubbing System

A gas scrubbing system was necessary to make sure the outgoing gases from the system were sufficiently clean before being released into the atmosphere. The gas flows out from the furnace tube and into an empty catch which is there to make sure that any back flow caused by a pressure gradient during cooling will not go back into the furnace. The gas then flows into a beaker containing water which allows any gases to flow through and create acids depending on what type of gaseous environment was used for the test. The next beaker contains a basic solution which will neutralize the acidic solution. Finally, the last beaker contains hydraulic fluid in order to catch any remaining contaminants from the gas before it is released into the ventilation system.

Furnace

One of the critical elements in the design of a high temperature electrochemical setup is the selection of a furnace. This furnace should be designed to suspend the electrodes and containment cell using a flange. The selected furnace was rated for a maximum temperature of 1200°C and an alumina tube was selected due to its refractory nature. The alumina was also selected because it would not react with different gases which may be flown through the system. A metal flange could then be used to suspend the three-electrode cell into the heat zone of the furnace. The flange was machined so that it could hold the crucible, electrodes, and gas lines in place. Additionally, the electrodes protruded from the flange with enough length for a potentiostat to be connected, thus allowing for electrochemical tests to be run.

Summary and Future Work

An experimental electrochemical test setup was designed and built to better observe high temperature corrosion phenomena. Future work will include using the setup to test molten salt corrosion behavior for various applications including nuclear energy, solar power, and industrial gas turbines.

References

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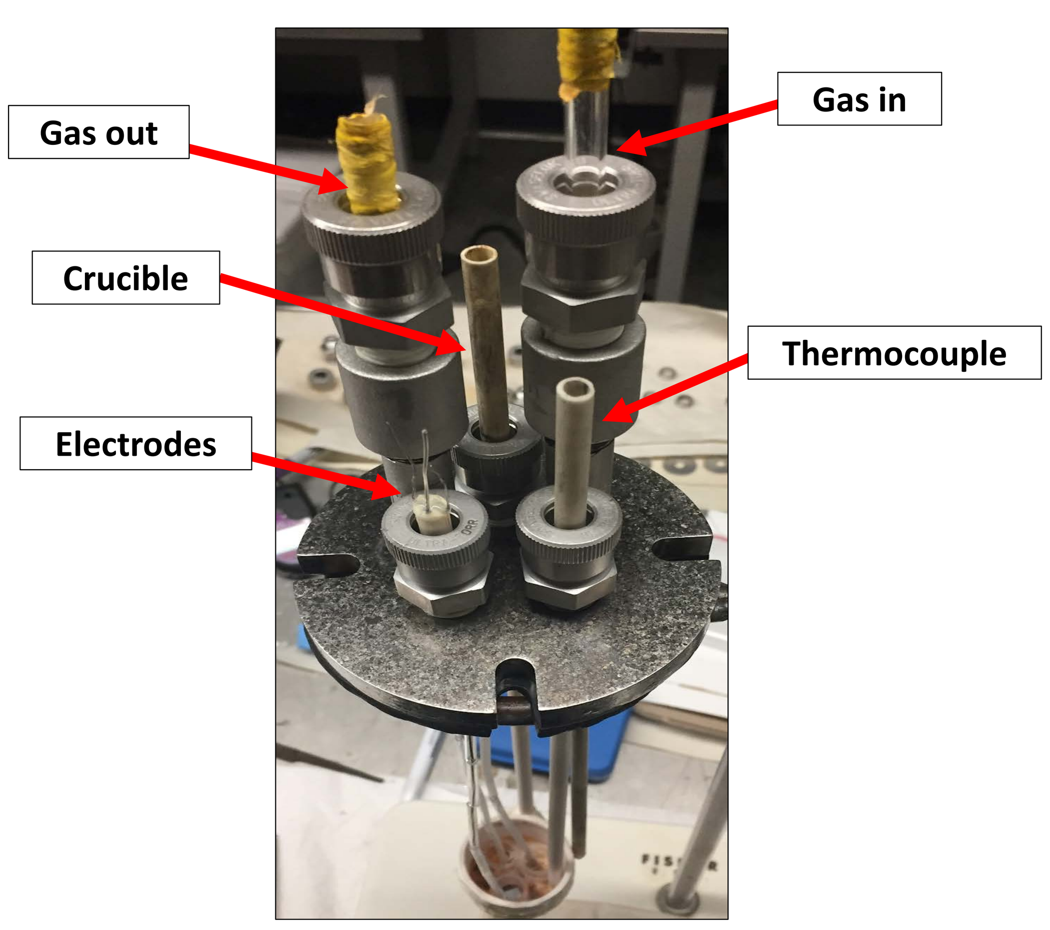
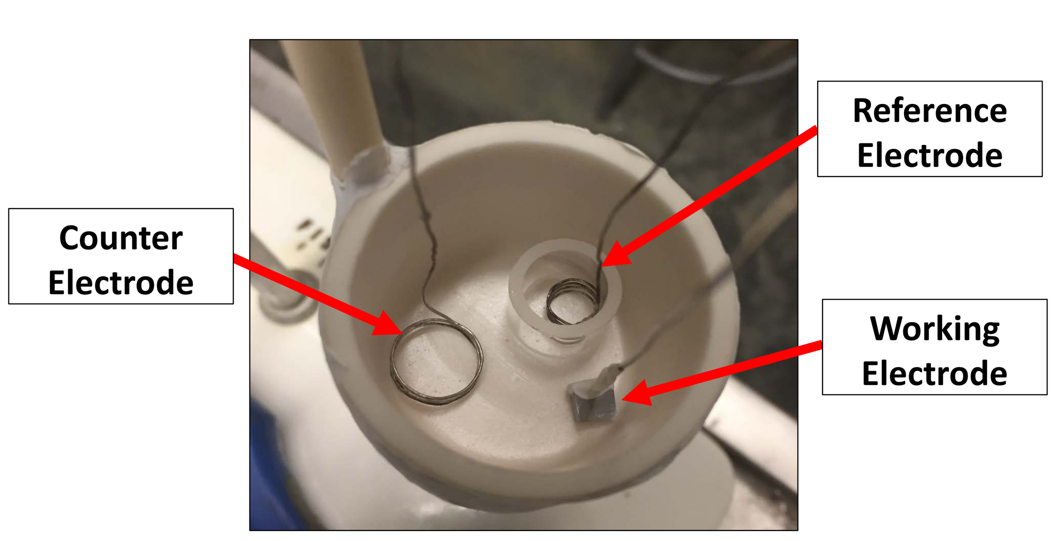


(A)



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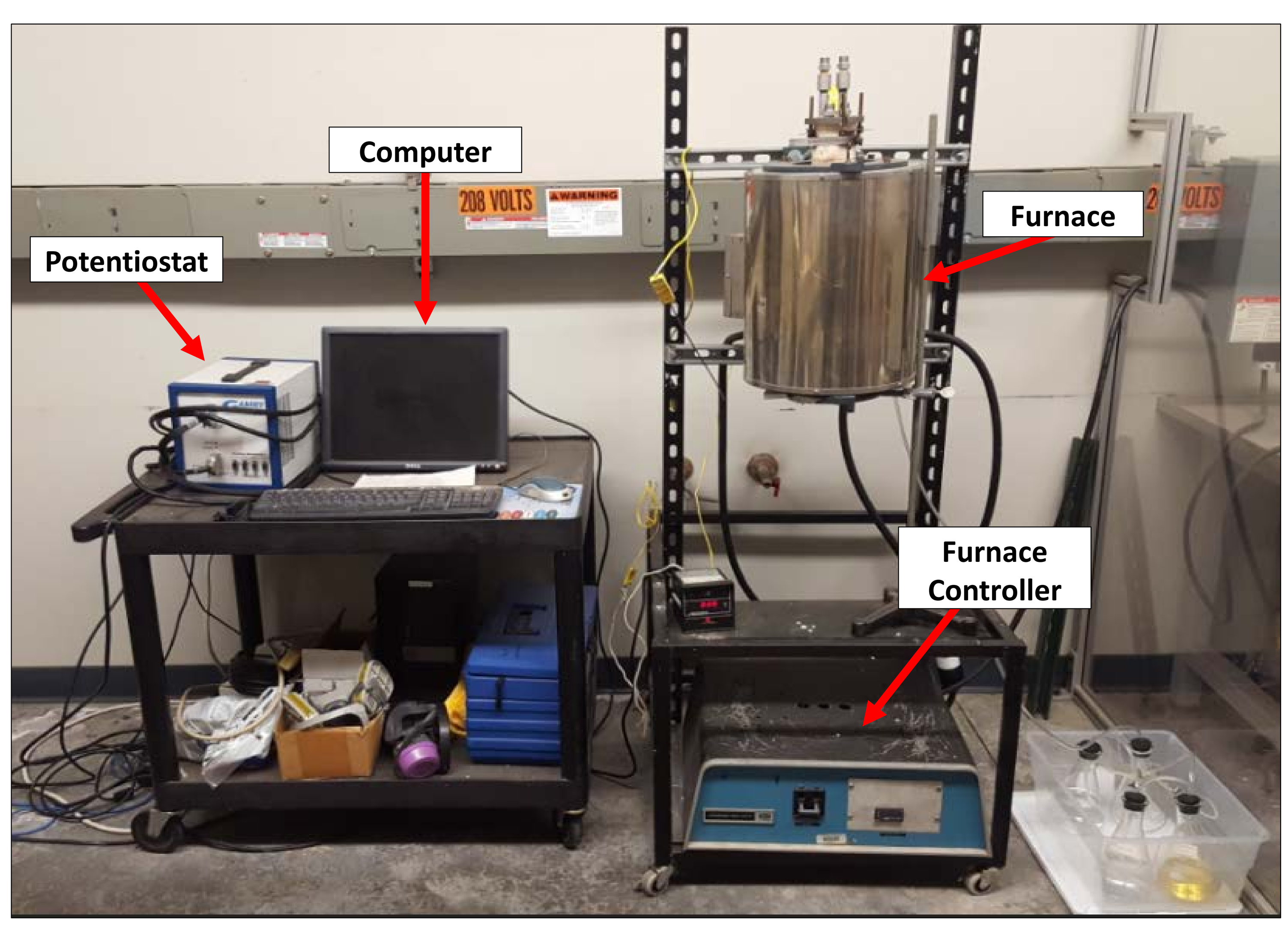

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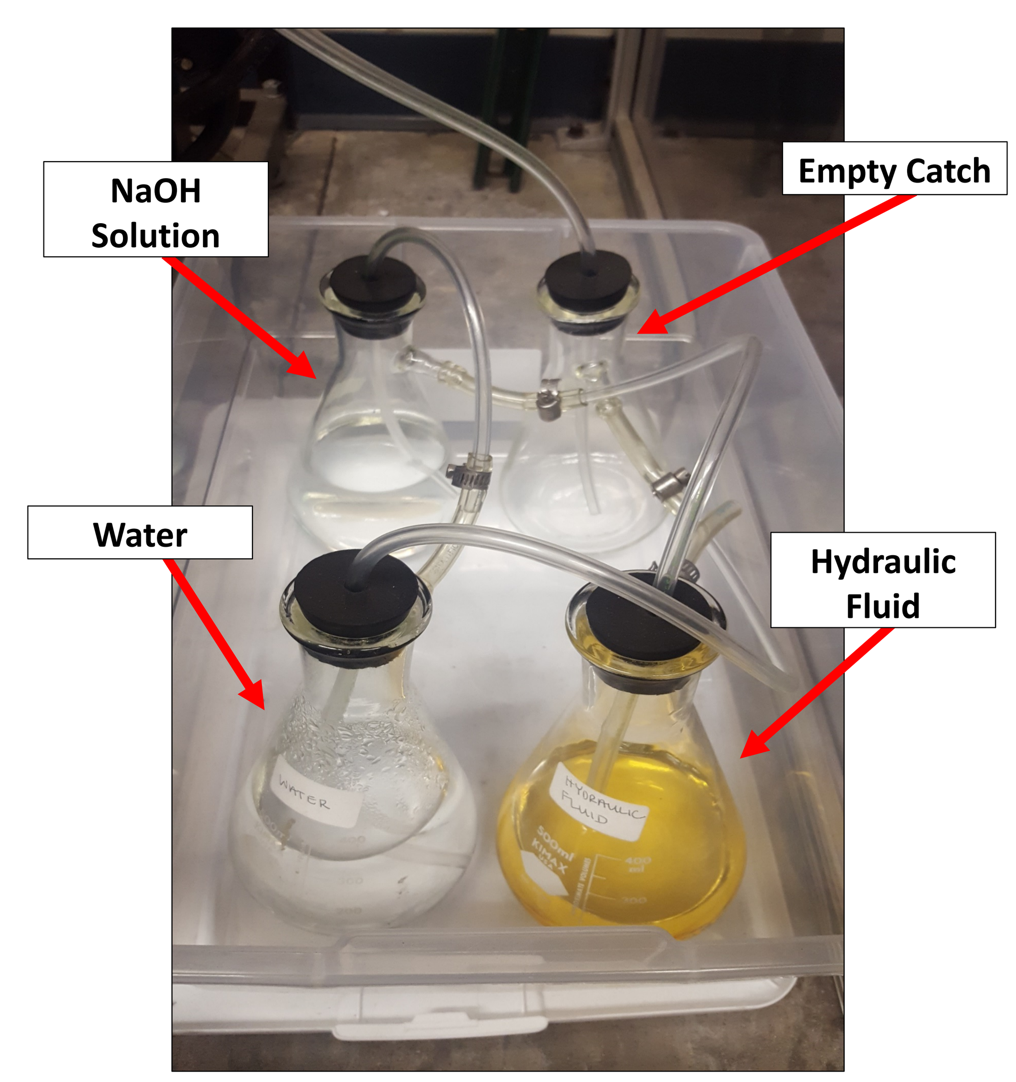

(D)

The figures in this section illustrate the experimental high temperature corrosion setup discussed above. Figure (A) is a detailed image of the flange design which holds all of the necessary components in place. This particular arrangement allows for the electrodes to have enough exposed area to be attached to the potentiostat. Figure (B) shows the three-electrode cell within the alumina crucible. The quartz sleeve around the reference electrode isolates the platinum wire from the bulk solution while still facilitating ionic transfer. (C) is a picture of the overall setup which encompasses the furnace, its controller, a computer, a potentiostat, and the gas scrubbing system. During testing, the potentiostat leads are hooked up to the electrodes and the data is recorded on the computer. Gas lines (not pictured) are also attached to the flange and the outgoing gases pass through the scrubbing system. Figure (D) shows the gas scrubbing system in detail. The purpose of the system is to neutralize any acidic gases which may have formed during testing.