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# System development for evaluating performance of corrosion resistant coatings exposed to molten copper chloride salt



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## ABSTRACT

Hydrogen, as one of the best alternative energy carrier candidates, is an important sustainable substitute for fossil fuels. Hydrogen demand has significantly increased as a clean fuel. To improve the green content of the future hydrogen economy, hydrogen must be produced in a clean method. The Cu–Cl cycle is one of the capable thermochemical cycles which is supposed to provide clean hydrogen production. Since this cycle has some high temperature stages, a study on these parts is essential to realize the entirety of the cycle, including the high temperature anti-corrosion equipment materials and the system to evaluate the performance of the materials.

In one of the high temperature steps, molten Cu–Cl is produced as an intermediate product, which brings in the challenges of equipment materials selection. This paper is about developing a system to evaluate candidate coatings following an immersion test in the molten Cu–Cl at 500 °C for a prolonged time (i.e. 4 h, 10 h, 48 h, and 100 h). This system was developed in order to run the experiments in safe manner as a fire/smoke/corrosion resistant coating.

Previous immersion test design was improved to perform an immersion test that mimics a similar condition at high temperatures with a commercial hydrogen plant. All safety procedures and safety mechanisms were developed and a new geometry was designed for samples. It was proved that this system was capable to evaluate the performance of coatings at high temperature for molten salts and the system could accommodate coating tests in a safe manner. Later, some ceramics with metallic coatings were applied and then analysed in diverse combinations to the base metal (medium carbon steel) using thermal spray methods.

The design and the performance of mechanism improvements are evaluated by measuring the corrosion rate. A study of weight change of the specimen on exposure, which is indicative of corrosion rate, represents the effectiveness of the developed sealing mechanism. The corrosion rate decreased and then approached to zero, as such sealing mechanism performed well under the experimental conditions.

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## Introduction

Hydrogen is considered to be one of the best alternative energy carrier candidates and is easier to sustain than fossil fuel. Since hydrogen is a copious chemical element, there are no concerns about running out of it. Hydrogen production demand has arisen in many major industries such as petroleum, gas, agriculture and transportation [1]. Particularly, hydrogen is proposed to be the most promising candidate for the future clean energy in the transportation industry [2].

Some methods for hydrogen production cycles being studied have better efficiency and can possibly be practical for commercial use [2]. These methods are based on using catalysts in a thermochemical cycle to dissociate hydrogen from water. The majority of hydrogen production cycles which are being considered, involve processes require temperature over 800 °C [2]. The high operating temperature will bring other challenging problems including plant design, fire hazard and material selection [3]. This method is based on using Copper Chloride as the catalyst, which is called Cu–Cl cycle, has several advantages compared to the other cycles being investigated, including a lower operating temperature [3]. The highest operating temperature in the Cu–Cl cycle is around 530 °C, which is much lower than highest temperature for other cycles [3].

The terms 'high-temperature corrosion' or 'hot corrosion' are normally taken to apply to the reactions taking place between metals and gases at temperatures above 100 °C. During the hot corrosion, the presence of a liquid electrolyte is not necessary. This type of damage is called *dry corrosion* or *scaling* [5]. It is obviously in contrast with cases of wet corrosion at temperatures above 100 °C, i.e. in pressurised boilers. Practically, some specific metals and alloys used at temperatures above normal do not suffer too much measurable attack in the atmosphere till the temperature is considerably above 100 °C. Some kind of metals such as iron and low-alloy steels form only the thinnest of interference oxide films at around 200 °C. Copper usually shows the first sign of tarnishing at around 180 °C, and while aluminium forms a thin oxide film at room temperature, the growth rate is extremely slow even around its melting point [4].

For the most significant practical hot-corrosion (to consider the passive fire engineering) phenomena, reactions are controlled by the kinetics of the proceeding, rather than by the thermodynamic stability of the reactants or products involved. It is considerable that reaction occurred under simplified laboratory conditions are inappropriate for the more complicated conditions in practice [6].

A wide variety of molten salts are used in industries having a wide range of possible chemical reactions with metallic portions. Molten salts mostly involve mixtures of carbonates, nitrates or alkaline metals or halides of the alkaline-earth and these usually have solubility for oxides of the other metals. (Therefore, the molten salts can react with other alloys to produce oxides). The produced oxides do not form a protective coating and are consequently move into solution of the molten salts and this attack steadily progresses. In some cases adsorption of components of the salt mixture into the alloy can take place, with either beneficial or deleterious results [6].

The only research had been published in this specific topic was done by Edwin Siantar [4]. Siantar designed and developed a system to carry out coating evaluations; however, none of the coating candidates tested by him survived the immersion test in which these were exposed to molten Cu–Cl at 500 °C.

Siantar's apparatus and samples physically provision helped our research group to conduct more precise studies on his method. After studying Siantar's research and his conclusions, as they indicate severe corrosion on some reliable candidates (even within short periods of time) and lack of integrity on the most of coatings' edges (it could be due to the non-uniform coating application on the sample sharp edges), it was decided to re-evaluate the entire experimental process and the specimen geometry. A bullet shape was chosen as the sample geometry to avoid lack of coating integrity on the sharp edges. The apparatus was also modified to accommodate the new geometry and to remove the sources of errors.

Later, some ceramics with metallic coatings were applied and then analysed in diverse combinations to the base metal (medium carbon steel) using thermal spray methods [14].

## Analysis/methodology

The corrosion testing apparatus was designed to evaluate the material performance in an environment similar to the proposed Copper Chloride (Cu–Cl) cycle hydrogen production. A practical method to simulate an environment for candidate materials was immersing samples in molten Cu–Cl at an elevated temperature (500 °C) for a prolonged period of time (more than 100 h) to evaluate the effects of exposure to molten Cu–Cl. This section explains the design requirements and material selections for an immersion apparatus. It goes on to describe the design, assembly, and operation of the immersion apparatus [4].

For a safe and reliable operation, the immersion test apparatus requirements are listed below;

1. The test vessel had to contain the molten Cu–Cl solution. The materials of vessel or any other container that were in contact with the molten Cu–Cl should be chemically inert, and they should not react with the molten Cu–Cl and Cu–Cl salt, and should have an operating temperature higher than 550 °C.
2. The immersion test vessel needed to be sealed completely in order to prevent any leakage and other gaseous impurities from entering the vessel. Main parts that required more attentions during the designing were the joints between the tubing and pipe connection, and the joint between lid and body of the vessel. Eliminating unwanted gas presence was one of the most important factors. Unwanted gas could interfere with the reaction during the sample exposure, which could affect the corrosion reaction. For instance, if oxygen was inside the vessel due to a leak, it could react with the surface of the material being tested. The reaction between oxygen and a metallic surface at high temperature could create an oxide layer as well as from water vapour. The surface condition of these samples

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