

**CORROSION STUDY OF STAINLESS STEEL  
ELECTRODES EXPOSED IN MOLTEN CARBONATE  
SALTS ELECTROLYSIS**

**AFENLEY INGGIU**

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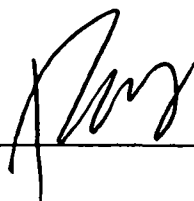
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## CERTIFIED BY

  
\_\_\_\_\_

**DR Nancy Julius Siambun**

**SUPERVISOR**



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## ABSTRACT

This study is mainly concerned with the corrosion attack on the 304 stainless steels on 3 type of voltage feeding; continuous, pulsing and alternate. Apart from that different type of ternary mixture of the molten salt;  $\text{Li}_2\text{CO}_3\text{-CaCl-LiCl}$  and  $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-Na}_2\text{CO}_3$  is used in carried out the result based on the objective. An attempt to investigate the corrosion of the electrode metal in  $\text{Li}_2\text{CO}_3\text{-CaCl-LiCl}$  molten salt and also in  $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-Na}_2\text{CO}_3$  molten salt was also made through the process electrolysis in 3 hours periods. An attempt to investigate and observe the microstructure of electrodes metal after the electrolysis process in the molten salts with various electrolysis voltage feeds also done. The experimental results were tabulated in form of table such as, weight loss in electrodes. The graphical results also plotted to observe the behaviour or pattern of each process electrolysis feeding. Result from data show that different type of molten salts used gives different result of images under optical microscope with different type voltage feeding. Weight loss of electrodes also shows differences in data obtained from the experiment conducted.



## ABSTRAK

*Kajian ini adalah berkaitan dengan serangan kakisan atau karat pada 304 keluli tahan karat terhadap 3 jenis elektrolisis voltan yang berlainan. Selain itu 2 jenis 3 campuran garam lebur;  $\text{Li}_2\text{CO}_3\text{-CaCO}_3\text{-LiCl}$  dan  $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-Na}_2\text{CO}_3$  digunakan dalam kajian ini keputusan berdasarkan objektif. Kajian untuk menyiasat kakisan logam elektrod dalam  $\text{Li}_2\text{CO}_3\text{-CaCO}_3\text{-LiCl}$  garam lebur dan juga di  $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-Na}_2\text{CO}_3$  garam lebur telah dibuat melalui elektrolisis proses dalam tempoh 3 jam. Kajian untuk menyiasat dan memerhati mikrostruktur elektrod logam selepas proses elektrolisis dalam leburan garam dengan pelbagai voltan elektrolisis juga telah dilakukan. Keputusan eksperimen direkodkan dalam bentuk jadual seperti beza jisim akhir elektrod. Keputusan grafikal dihasilkan untuk membezakan setiap proses elektrolisis. Keputusan dari data menunjukkan bahawa jenis garam lebur yang berbeza digunakan, memberikan hasil yang berbeza melalui analisis imej di bawah mikroskop optik dengan process elektrolisis yang berbeza. Perubahan jisim electrod dari kajian ini dijelaskan dalam data yang diperolehi daripada eksperimen yang dijalankan*



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# CHAPTER 1

## INTRODUCTION

An electrode is a conductor that passes an electrical current from one medium to another, usually from a power source to a device or material it including a wire, a plate, or a rod, and is most commonly made of metal, such as silver, lead, or but can also be made of a non-metallic substance that conducts electricity, such as graphite. According to Obreja (2008) there is various kind of electrode that previous research as electrode for example nickel, chromium, copper, iron was used for electro-chemical process as such production of aluminium, purification of titanium, electro-carburisation process, production of carbon, and many more. Very often it is reported that the electrodes either anode or cathode that were connected to terminal positive and negative respectively in the electrolysis process suffered from corrosion and degrading the electrode function as electrodes.

According to Gribaudo and Rameau (1983) molten salt system usually operates at higher temperature than in aqueous solution, corrosion in molten salts is usually much faster and producing thick scale in molten salt corrosion.

The studies of the corrosion could affect societal issues that include human life and safety, the cost of corrosion, conservation of materials. The study of corrosion involves intersection of chemistry and material science and mechanic. The



science of mechanics must be added to understand mechanically assisted corrosion process such as stress corrosion, cracking and corrosion fatigue. The challenges for engineer and scientist for this corrosion studies is the formulation the new generation of stainless steel to replace others metals

### 1.1 Problem statement

In the molten salt electrochemical process (Wang, 2012) (Chen, 2012) stainless steel metal was one type of material used as anode and cathode electrodes in high temperature molten salt containing chloride and carbonate element. Molten salt is very corrosive especially in very high electrolysis temperature i.e.  $>500^{\circ}\text{C}$ . The studies of the effects of high molten salt temperature and type of molten salt used in the electrolysis process to produced carbon had never been carried out. Therefore there is a need to carry out corrosion study, to further understand the effects towards a sustainable electrode for molten salt electrolysis.

In this work, corrosion of electrode exposed in two type of molten salt i.e.  $\text{Li}_2\text{CO}_3$ - $\text{CaCO}_3$ - $\text{LiCl}$  and  $\text{Li}_2\text{CO}_3$ - $\text{K}_2\text{CO}_3$ - $\text{Na}_2\text{CO}_3$  will be focus.

### 1.2 Objectives

The purpose of this research project is to study the effect of the molten salts electrolysis on the stainless steel as the sustainable electrode. The experiment or test is conduct at two different carbonate molten salts;  $\text{Li}_2\text{CO}_3$ - $\text{CaCO}_3$ - $\text{LiCl}$  and  $\text{Li}_2\text{CO}_3$ - $\text{K}_2\text{CO}_3$ - $\text{Na}_2\text{CO}_3$  molten salt. The test is exposes with higher temperature for the corrosion behaviour to occur in the electrodes. By the observation of the microstructural on the electrode for both anode and cathode terminal in optical microscope after the test is can be compared between the two mentioned molten carbonate salts.

The objectives of this thesis are;

- A. To study the corrosion of the electrode metal in  $\text{Li}_2\text{CO}_3\text{-CaCO}_3\text{-LiCl}$  molten salt and also in  $\text{Li-K-Na}_2\text{CO}_3$  molten salt through the process electrolysis.
- B. To study the microstructure of electrodes metal after the electrolysis process in the molten salts with various electrolysis voltage feed

### 1.3 Scope of work

This project is to study the corrosion of the electrodes in molten carbonates salt electrolysis. The loss in weight of the electrode is one of the parameter of the corrosion to occur. From the two different molten salts electrolysis proses the behaviour of the corrosion can be compared to. To analysis the corrosion studies there the aspect as weight is need to take as consideration. By observation of microstructural of the cleaned electrodes (stainless steel) the analysis of the corrosion study can be done.

The experiment is conduct in 3 stages the stages is including continuous electrolysis process, pulsing and alternate terminal electrolysis process. First stages is by conducting continuous electrolysis of molten salts in the heat chamber, the molten salts with the stainless steel as the electrodes is being heated in the heat chamber. The requirement of this process is conduct at 550 with 4volts of voltage. This process is conducted in 3 hours where these periods of time the behaviour of the corrosion is can be achieve. Next the pulsing process is continued and also the alternate terminal electrolysis process. After the three stages are done or complete the electrode metal is taken out, then measure the weight of the electrode metal. But measure the initial weight too. Then the electrode is cleaned observed the microstructural of electrodes by using optical microscope.



#### **1.4 Research benefit/expected outcome**

The advantageous of this research is the corrosion failure can be study from the microstructural of the electrodes, so that the failure that due to the corrosion can be minimized. Economic losses can be reduced as the cost of replacing corroded structure can be minimized.

## **CHAPTER 2**

### **LITERATURE REVIEWS**

#### **2.0 Introduction**

In this topic is discussed about the general knowledge in molten salt electrolysis. Some of knowledge on the type of the stainless steel used also reviewed. Since this study is more about corrosion. So there is some factor that lead to corrosion is also discussed. The behaviour of corrosion in high temperature is also thing that reviewed on this chapter. Most important is deposition of carbon that leads to corrosion behaviour also discussed.

#### **2.1 Molten salts electrolysis**

To study the corrosion in the presence of molten salts, the molten salts analysis is needed to act as a medium for the corrosion to occur. In this electrochemical setup of molten salts is prepare and electrode also prepared. This electrode is act as conductor that passes electrical current from one medium to another. The electrodes of a battery are separated by a solution containing ions (electrically charged atoms or groups of atoms). One of the electrodes—the negative electrode—undergoes a chemical reaction that gives it excess electrons. The other electrode—the positive electrode—undergoes a chemical reaction that removes electrons. When the two electrodes are connected by an external electrical circuit, the excess electron flow from the negative to positive electrode

Electrolytic cells and electron tubes are connected to an external source of electrical power, such as a battery or dynamo. The power source moves electrons into one of the electrodes (making it the negative electrode), withdraws electrons from the other (making it the positive electrode), and causes a current to flow through the medium between them.

The electrode through which negative charge enters an electrical device is called the cathode; the electrode through which negative charge leaves is called the anode. The negative electrode of a battery is thus the anode, whereas the negative electrode of an electrolytic cell is the cathode

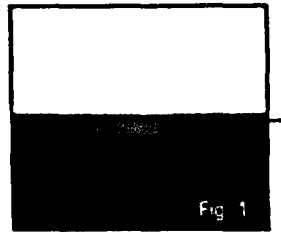
An electrode material to be used successfully in severely corrosive conditions such as in the electrolysis of molten halide salts and particularly of molten fluoride salts should primarily be chemically and electrochemically stable at the operating conditions. It should also be catalytic with respect to the anodic evolution of oxygen and/or halides, so that the anode over potential is lowest for high overall efficiency of the electrolysis process. The electrode should also have the thermal stability at operating temperatures of, i.e., about 200° to 1100° C., good electrical conductivity and be sufficiently resistant to accidental contact with the molten metal cathode.

The molten salts should exhibit a wide range of electro-inactivity; its decomposition voltage should be large. It should contain ionic species with higher discharge potential (oxidation potential) than the metal ion of metal salts being electrolyzed (cation) and the anion of the salts (anion). The solvent should be more stable electrochemically than the metal salt being electrolyzed and have tendency to deposit. The voltage is applied into the molten salts so that the electrolysis process can be proceeds.

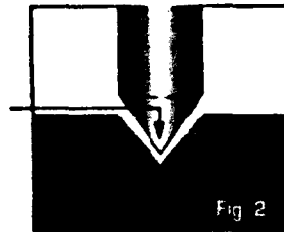
## 2.2 Stainless steel properties

In 1913, English metallurgist Harry Brearly, working on a project to improve rifle barrels, accidentally discovered that adding chromium to low carbon steel gives it stain resistance. In addition to iron, carbon, and chromium, modern stainless steel may also contain other elements, such as nickel, niobium, molybdenum, and titanium. Nickel, molybdenum, niobium, and chromium enhance the corrosion resistance of stainless steel. It is the addition of a minimum of 12% chromium to the steel that makes it resist rust, or stain 'less' than other types of steel.

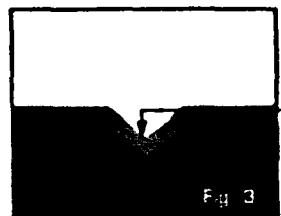
The chromium in the steel combines with oxygen in the atmosphere to form a thin, invisible layer of chrome-containing oxide, called the passive film. The sizes of chromium atoms and their oxides are similar, so they pack neatly together on the surface of the metal, forming a stable layer only a few atoms thick. If the metal is cut or scratched and the passive film is disrupted, more oxide will quickly form and recover the exposed surface, protecting it from oxidative corrosion. (Iron, on the other hand, rusts quickly because atomic iron is much smaller than its oxide, so the oxide forms a loose rather than tightly-packed layer and flakes away.) The passive film requires oxygen to self-repair, so stainless steels have poor corrosion resistance in low-oxygen and poor circulation environments. In seawater, chlorides from the salt will attack and destroy the passive film more quickly than it can be repaired in a low oxygen environment.



**Figure 2.1:** In any normal oxidising environment a protective coating of passive chromium rich oxide film is automatically formed on stainless steel.



**Figure 2.2:** When scratched, damaged or machined this protective film is denuded exposing the steel to the atmosphere.



**Figure 2.3:** The protective coating is quickly restored through the rapid self-repairing quality of the chromium rich film.(source:Australian Stainless Steel Development Association (ASSDA))

The main reason of existence of stainless steels is their resistance toward corrosion. The main chemical composition or alloy element is chromium it should be at least contain 11% in stainless steel because it is reactive element. Other alloy element such as manganese, nickel, carbon and nitrogen makes the stainless steel more resistance in different environment. The stainless steel behaviour in corrosion resistance, mechanical properties and cost is varying over broad range. Therefore it is important to specify suitable type of stainless steel for different application.

### **2.3 Type of stainless steels**

Stainless steel is divided into many types according to the composition of the different type of stainless steel. The type of stainless steel that being discussed on this topic is Austenite stainless steel, Ferritic stainless steel, Duplex stainless steel, Martensite stainless steel and Precipitation hardening stainless steel. These differences in properties on each type of the stainless steel could bring different type of corrosion to occur.

#### **2.3.1 Austenite stainless steel**

Stainless steel that added with nickel in sufficient amounts the crystal structure is called "austenite". The basic composition of austenitic stainless steels is 18% chromium and 8% nickel. Austenitic steels have austenite as their primary phase (face centered cubic crystal) and it also not hardenable by heat treatment. Austenitic grades are the most commonly used stainless steels accounting for more than 70% of production.

#### **2.3.2 Ferritic stainless steel**

Ferritic steels have ferrite (body centered cubic crystal) as their main phase. These steels contain iron and chromium, based on the Type 430 composition of 17%

chromium. Ferritic steel is less ductile than austenitic steel and is not hardenable by heat treatment.

### **2.3.3 Martensite stainless steel**

These steels are similar to ferritic steels in being based on Chromium but have higher Carbon composition. This allows them to be hardened and tempered much like carbon and low-alloy steels. They are used where high strength and moderate corrosion resistance is required. Martensitic steels are low carbon steels which are containing composition of 12% chromium, and 0.12% carbon. Martensitic gives steel great hardness, but it also reduces its toughness and makes it brittle, so few steels are fully hardened.

### **2.3.4 Duplex stainless steel**

These steels have a microstructure which is approximately 50% ferritic and 50% austenitic. This gives them a higher strength than either ferritic or austenitic steels. They are resistant to stress corrosion cracking. Lean duplex steels are formulated to have comparable corrosion resistance to standard austenitic steels but with enhanced strength and resistance to stress corrosion cracking.

“Superduplex” steels have enhanced strength and resistance to all forms of corrosion compared to standard austenitic steels. They are weldable but need care in selection of welding consumables and heat input. They have moderate formability. They are magnetic but not so much as the ferritic, martensitic and PH grades due to the 50% austenitic phase.

### **2.3.5 Precipitation hardening stainless steel**

These steels can develop very high strength by adding elements such as Copper, Niobium and Aluminium to the steel. With a suitable “aging” heat treatment, very fine particles form in the matrix of the steel which imparts strength. These

steels can be machined to quite intricate shapes requiring good tolerances before the final aging treatment as there is minimal distortion from the final treatment. This is in contrast to conventional hardening and tempering in martensitic steels where distortion is more of a problem. Corrosion resistance is comparable to standard austenitic steels like 304.

### **2.3.6 Austenite stainless steels properties**

In this study of corrosion type of stainless steel is chosen as electrodes is austenite 304 stainless steel. This is due to this type of stainless steel is mainly use in many sector and it commonly used for more than 70% production. This kind of austenite stainless steel also highly used in mechanical components such as in higher temperature sector such as steam generator. These 304 stainless steels are complex alloys that made from iron and also contain higher chemical composition of Chromium. This Chromium is important layer that preventing the corrosion to occurs. Consequently, applications of austenitic steels are restricted to higher temperature applications and specific situations where severe corrosion conditions occurred. (Ishak 2008).

Beside that austnite 304 stainless steels have face centered cubic structure this makes the stainless steel more stable structure and have better performance or property of strength when exposed in higher temperature of 550°C-770°C. the structure is really stable structure because of the chemical composition of manganese, nickel and nitrogen that exist in this type of austenite stainless steels. The advantages of this austenite 304 stainless steel are high resistance in corrosion, high toughness at higher and extreme temperature.



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