

**SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
UNIVERSITI SAINS MALAYSIA**

**DEVELOPMENT OF DIFFUSION BARRIER-FORMING COATING FOR
HIGH-TEMPERATURE APPLICATION: EFFECT OF COATING
DURATION AND BATH TEMPERATURE**

By

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Dissertation submitted in partial fulfillment
of the requirements for the degree of Bachelor of Engineering with Honours
(Materials Engineering)

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled “**Development Of Diffusion Barrier-Forming Coating For High-Temperature Application: Effect Of Coating Duration And Bath Temperature**”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

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LIST OF SYMBOLS

A	Ampere
wt. %	Weight Percentage
h	Hour
<	Less than
°	Degree
α	Alpha
Θ	Theta angle
cm	Centimeter
°C	Degree Celsius
Cm ²	Centimeter square
μm	Micrometer

LIST OF ABBREVIATIONS

$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$	Ammonium Heptamolybdate
$\text{C}_2\text{H}_4\text{O}_2$	Concentrated Acetic Acid
$\text{C}_2\text{H}_7\text{NO}_2$	Ammonium Acetate
CH_3COOK	Potassium Acetate
CVD	Chemical Vapor Deposition
EDX	Energy Dispersive X-Ray
FESEM	Field Emission Scanning Electron Microscope
HCl	Hydrochloric acid
HER	Hydrogen evolution reaction
HNO_3	Nitrate acid
ICDD	International Centre for Diffraction Data
OCP	Open circuit Potential
OER	Oxygen evolution reaction
PVD	Physical vapor deposition
SS304	Stainless Steel 304
XRD	X-ray Diffraction
XRF	X-Ray Fluorescence

PEMBANGUNAN LALUTAN PEMBENTUK PENGHALANG RESAPAN
UNTUK APLIKASI SUHU TINGGI: KESAN TEMPOH LALUTAN DAN SUHU
MANDI

ABSTRAK

Pembakaran biojisim akan membebaskan klorin, kalium, sulfur, nitrogen, dan silikon. Kehadiran kandungan klorin dan alkali yang tinggi daripada bahan api biojisim boleh menyebabkan kerosakan teruk pada unit pembakaran. Kebimbangan terbesar bahan pemanas lampau dalam loji biojisim ialah isu pengaratan semasa pembakaran biojisim suhu tinggi. Interdifusi antara salutan dan bahan telah menjejaskan jangka hayat pemanas lampau. Untuk menyelesaikan masalah ini, salutan penghalang telah dicadangkan untuk mengurangkan interdifusi unsur salutan ke bahan dinding pemanas lampau. Molibdenum dipilih sebagai elemen salutan kerana sifatnya yang sangat baik. Molibdenum sangat sukar disalut pada permukaan keluli tahan karat. Dalam kajian ini, kesan julat suhu mandian antara 30-35 °C, 40-45 °C, dan 50-55 °C dan tempoh salutan yang berbeza iaitu 1 jam, 2 jam dan 3 jam telah dikaji untuk menentukan suhu optimum dan tempoh salutan untuk pembentukan salutan molibdenum padat pada permukaan keluli tahan karat 304. Berdasarkan morfologi permukaan dan analisis unsur dan luas keratan rentas, kedua-dua hasil pencirian telah membuktikan kehadiran molibdenum pada keluli tahan karat pada suhu dan tempoh yang berbeza. Hasil analisis unsur telah mengesan peratus molibdenum tertinggi pada 40-45 °C selepas pendedahan 3 jam.

DEVELOPMENT OF DIFFUSION BARRIER-FORMING COATING FOR HIGH-
TEMPERATURE APPLICATION: EFFECT OF COATING DURATION AND
BATH TEMPERATURE

ABSTRACT

During the combustion of biomass, it will release chlorine, potassium, sulfur, nitrogen, and silicon. The presence of a high concentration of chlorine and alkali content from biomass fuels can cause severe damage to the combustion units. The biggest concern of the superheater material in biomass plants is the issue of corrosion during high-temperature combustion of biomass. The interdiffusion between coating and the material has affected the lifespan of the superheater. In order to solve this issue, it is suggested to apply a barrier coating to mitigate the interdiffusion of the coating elements to the material of the wall of the superheater. Molybdenum is chosen as the coating element due to its excellent properties. Molybdenum is very difficult to coat on the surface of stainless steel. In this study, the effect of bath temperature ranges between 30-35 °C, 40-45 °C, and 50-55 °C and different coating duration which is 1 hour, 2 hours and 3 hours were studied to determine the effect of temperature and coating duration towards molybdenum coating on the surface of stainless steel 304. The surface of coating was analysed using FESEM with EDX and XRD. Based on the surface morphology and elemental analysis and the cross-sectional area, both of the characterization result had proved presence of molybdenum on stainless steel at different temperature and duration. Result of elemental analysis had detect highest percent of molybdenum at 40-45 °C after 3 hour deposition.

CHAPTER 1 INTRODUCTION

1.1 Introduction

Today, the most potential energy source for lowering greenhouse gas emissions is biomass. Previously, fossil fuels had dominated the world energy suppliers for decades. Figure 1.1 shows that fossil fuels had contributed approximately 80% of the total world energy consumption. Carbon dioxide is produced during the burning of fossil fuels. The presence of carbon dioxide (CO₂) has brought negative effect to the surrounding such as pollution and global warming. Due to the increase in the demand for electrical energy, numerous industrial emit high amount of CO₂ through high rate of fossil fuel combustion. Several processes in industrial such as product of mineral products and metal such as iron also produce CO₂ emissions through chemical reactions even though the process do not involve combustion (Chen et al, 2015).

When biomass is burned, a number of elements are released, including chlorine, potassium, sulphur, nitrogen, and silicon. Chlorine and alkali from biomass fuels can seriously harm the combustion units when present in large concentrations. The main issue facing biomass facilities is corrosion of superheater tubes during high temperature brought on by corrosive element on the surface. Chloride have the ability to accelerate the movement of inorganic compounds such as potassium. Potassium chloride is the stable alkali-containing gas during high temperature. Alkali hydroxides is stable gas-phase species when it exposed to certain moisture when there is no chlorine present. (Khan et al, 2009).

High inlet temperatures are directly related to an enhancement in thermal efficiency in hot temperature applications, which raises the thermal loads placed on the materials used to make turbine components. When materials are used at high temperatures, it will lead to oxidation and corrosion. During high-temperature

combustion, the metals alloys in the combustion chamber easily oxidized when there is a lot of air present (Xu et al, 2008). Protective coatings are the most frequently employed corrosion mitigation strategy to prevent further oxidation and corrosion of the material (Chellaganesh et al, 2021). The presence of KCl significantly increases the risk of corrosion of stainless steel during high temperature application, especially in biomass power plants with huge power capacities and steam temperatures above 540 °C.

Interdiffusion of elements in the coating occurs during high-temperature applications. The interdiffusion of elements has caused the change in the composition of the original coating. Chromium-rich oxide formed during the stainless steel is used as the wall of the superheater. Generally, chromium oxide acts as the protective layer on stainless steel. However, the chromium oxide layer may break and dissolve in molten salt due to the presence of high potassium (K) and chlorine (Cl) from the product of combustion of biomass. The presence of corrosive elements increased the rate of corrosion. According to the research work of *Mudgal*, the oxide dissolution in the molten salt caused a pitting attack on the wall of the superheater (Mudgal et al., 2014). In order to prevent the interdiffusion of the oxide layer to the material of the superheater.

To prevent the interdiffusion of the coating towards the substrate, a diffusion barrier needs to be developed and applied on the surface of the substrate. The development of diffusion barriers on the substrate is the main focus of this research work. A good coating should consider the thermodynamic stability of the phases that constitute the coating as well as a proper deposition method. In order to get a good corrosion protection at high temperature, the coating microstructure must consists of low porosity. A dense coating microstructure can eliminate the diffusion of corrosive elements toward the substrate whereby the interdiffusion layer will avoid the diffusion

of coating elements into the substrate which could decrease the mechanical properties of the alloy. Diffusion coatings can be developed via chemical vapor deposition (CVD), and physical vapor deposition (PVD). In this research work, the diffusion barrier will be produced via the electroplating method.

Stainless Steel 304 (SS 304) is a form of austenitic stainless steel that has outstanding weldability, high mechanical strength, and corrosion resistance. Stainless steel 304 has been used in a variety of industrial and non-industrial fields. When exposed to a severely corrosive environment, SS 304 can also corrode when there is presence of corrosive element (Saefuloh et al, 2020). Stainless steel material can reduce corrosion rate. To prevent high-temperature corrosion and increase corrosion resistance, stainless steel is coated with metallic coatings such as molybdenum on the surface of the material (Riastuti et al, 2018).

Molybdenum is selected as the candidate for coating material due to its excellent properties. Melting point of metallic molybdenum (Mo) is around 2623 °C. Molybdenum have a low thermal expansion coefficient so that it would not have large volume change. Good thermal shock resistance, and wear resistance is also one of the advantages of molybdenum. Those excellent properties making it a good option for high-temperature applications in the automotive industry, space applications, and other fields. Molybdenum coatings are typically applied to materials to boost their thermal and wear resistance (Syed et al, 2015). Normally, the thermal spray method is used to coat metallic coating on the material of high-temperature application due to it can increase resistance to abrasive and high-temperature conditions, corrosion, and wear. Molybdenum coatings were fabricated through thermal spraying process. The coatings that are fabricated through thermal spraying have excellent properties such as very good wear resistance, adhesion strength, and very low porosity. Besides, the coating had a

very high hardness and a smooth surface (Tailor et al, 2018). Therefore, the thermal spray method is one the best method to coat metallic coating.

In this research work, the electroplating method was selected to coat molybdenum on stainless steel 304 due to some advantages. One of the easy and cheap methods for applying coatings to a variety of substrate materials is electrodeposition (Syed et al, 2015). Electroplating is also known as electrodeposition. Electrodeposition is one of the techniques for metal and metallic alloy coating. The coating can be electrodeposited by the simultaneous reduction of two or more metallic cations present in an electrolyte solution. There are many parameter that would affect the condition of the coating such as bath temperature, current density, type of electrode and pH value.

The phases of the coating layer must be thermodynamically stable at the operating temperature in order to create a good diffusion barrier coating. To prevent early coating failure, interdiffusion between the coating layer and substrate should occur as slowly as feasible. In order to prevent thermal stresses during heating or cooling cycles, the thermal expansion coefficients of the substrate, coating, and oxide layer should also be as similar as feasible. Besides, the protective layers should be slow-growing to keep the coating reservoir depletion rate by the surface reaction at a low level. Additionally, a dense and flat splat structured coating is desired to enhance corrosion resistance at high temperatures because corrosive species usually diffuse through pores and along splat boundaries (Sidhu et al., 2006).

1.2 Problem Statement

The biggest concern of the superheater material in biomass plants is the issue of corrosion during high-temperature combustion of biomass. High-temperature corrosion had caused a series of problems to the biomass plant such as the lifespan of the superheater material. During the combustion of biomass, it released different types of

corrosive elements such as chlorine, and sulphur. The major problem of the superheater material is the deposit of chloride. In order to prevent the corrosion of high-temperature applications, a coating is needed to act as a protective layer to prevent the direct contact of the aggressive element with the substrate.

A proper material selection and a good barrier coating for superheater tubes is needed to mitigate the high-temperature corrosion of materials. As stated by Grégoire et al. (2020), both Fe-based and Ni-based alloys are material candidates for power plant applications. Today, material for superheater tubes has been replaced from conventional low alloy steel with austenitic stainless steel (FeCrNi-base alloy) due to its superior resistance to high-temperature corrosive environments (Scapin et al., 2014; Yuan et al., 2016). 304 stainless steel is a type of FeCrNi alloy, which was categorized in an austenitic stainless steel group that contained an acceptable amount of Cr and Ni.

Currently, the interdiffusion between coating and the material has affected the lifespan of the superheater. In order to solve this issue, it is suggested to apply a barrier coating to mitigate the interdiffusion of the coating elements to the substrate which is the material of the wall of the superheater. The requirement of barrier coating should have a high melting point. Besides, the barrier coating cannot have any phase transformations in thermal cycling at high temperature. As a diffusion barrier, low thermal conductivity and proper thermal cycling resistance are the one of the important requirement. Diffusion barrier must have a thermal expansion coefficient that corresponds to that of substrate materials. Resistance to corrosion and low sinterability of porous microstructures improve the ability to withstand corrosion (Lakiza et al, 2018).

Molybdenum is chosen based on its properties such as high melting point, low thermal conductivity, and low thermal expansion coefficient. Based on the previous

research, refractory metal such as molybdenum is very difficult to coat on stainless steel from aqueous electrolyte due to their properties. However, there are still some researchers who coated successfully through highly concentrated acetate-based aqueous electrolyte near room temperature for certain duration. The researcher had develop molybdenum coating on copper substrate via electrodeposition method for certain condition but there is no research of molybdenum coating on stainless steel 304 (Syed, 2015). Therefore, the condition of the bath variables such as bath temperature and coating duration was studied in this research to find the optimized duration and bath temperature in order to get a dense molybdenum coating.

Currently, there is lack of research on the electrodeposition of molybdenum on stainless steel 304 under certain conditions. The presence of a dense coating of molybdenum on the surface of stainless steel 304 and the effect of bath temperature and duration of the coating has not been studied. In this study, the electroplating process was undergone at different bath temperature ranges between 30-35°C, 40-45°C, and 50-55°C to determine the optimum temperature for the formation of molybdenum coating on the surface of stainless steel 304. In addition, the effect of the duration range of the coating process for 1 hour, 2 hours and 3 hours was also investigated in this study to get the optimum duration of the coating process to get a dense molybdenum coating on the surface of Stainless Steel 304.

1.3 Research Objectives

The objective of the project as follows:

- a) To develop a molybdenum (Mo) coating via electroplating method on the substrate.
- b) To investigate the effect of bath temperature and coating duration on the coating.

1.4 Scope of the study

The electrodeposition of molybdenum on the surface of stainless steel 304 under different conditions was investigated. In this project, the stainless steel 304 (cathode) and a platinum electrode (anode) will be put in an acetate electrolyte solution and connected with a potentiostat with a fixed temperature of 40-45 °C for 1 hour, 2 hours and 3 hours at 0.5A. Besides, the stainless steel 304 (cathode) and a platinum electrode (anode) will be put in an acetate electrolyte solution and connected with a potentiostat at 30-35 °C, 40-45 °C and 50-55 °C for 1 hour. In the development of the molybdenum diffusion barrier, the optimum duration, and bath temperature were chosen from the surface morphology thickness of the oxide layer and the phase present in the oxide layer. The properties of the phase of the oxide layer that formed such as surface morphology, elemental distribution and phase identification will be characterized by using Scanning Electron Microscope (SEM), Energy Disperse X-ray (EDX), and X-ray Diffraction (XRD).

1.5 Thesis outline

In this thesis, there is a total of five chapters. The first chapter is about to introduce the background, the objectives of this project, the problem statement and the research scope of the project. Chapter two is all about the literature review of the background relevant to the present work, including the high-temperature corrosion, electroplating method, stainless steel 304 and the material for high-temperature application. Chapter three of the thesis includes the detail of the procedure for preparation of sample and electrolyte solution, the overall experimental procedure and the characterization method used in this project. Chapter four of this thesis is to list and discuss the result of the experiment and comprehensive discussions on the development

of the molybdenum diffusion barrier on stainless steel 304. The result and discussion on the effect of the duration of coating and the effect of the bath temperature are included in chapter four. Lastly, the conclusion and recommendations are presented for further studies.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Fossil fuels are commonly used to generate energy worldwide. Through the combustion of fossil fuels, greenhouse gases generated had bring a few negative environmental impacts to the world. The release of greenhouse gases during combustion such as carbon dioxide and other gases had contributed to air pollution and global warming. Global warming and air pollution had become the major concerns around the world. Air pollution affected the quality of life of the population. Currently, many countries had found that renewable energy is a key issue to decrease fossil fuel usage and the shift to low carbon energy systems (Martins et al., 2019).

Currently, fossil fuels had replaced by biomass energy due to some reasons. Biomass has a different chemical composition compared to fossil fuels. Biomass has the ability to can recapture the CO₂ during combustion in a shorter time (Okoro et al., 2015). The combustion of fossil fuels releases carbon dioxide and other greenhouse gases which will trap heat in the atmosphere, whereas biomass is considered carbon-neutral energy. This fuel source had disastrous environmental consequences, ranging from air pollution to global warming.

During the combustion of biomass, the combustion involves a lot of heat. In biomass plants, the wall of the superheater tubes is exposed to hot and corrosive gas. For example, chlorine, sulphur, alkali metals, and oxygen will affect chlorine-based corrosion. Chlorine-based corrosion is a serious problem and it will degrade the work-life of the superheater tubes.

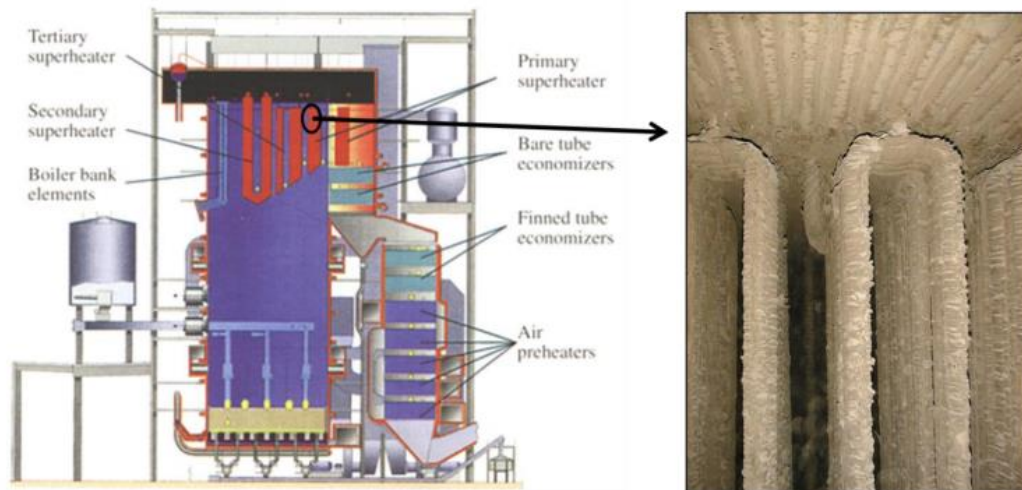


Figure 2.1 Schematic diagram of a biomass boiler highlighting the fly ash deposition in superheater tubes (Courtesy: Kvaerner Pulping)

In order to solve this problem, a coating is developed on the surface of the components. There are many types of coating techniques that had been introduced and proposed by many researchers. Literature studies in these fields especially on the coating techniques, the parameter of the electroplating method, and corrosion behaviour of superheater materials are important to know and discussed in the following section.

2.2 Biomass plant

Based on research work by *Teh*, fossil fuel is still the main source worldwide of energy consumption. Fossil fuel has brought a critical crisis with environmental consequences to the world. The combustion of fossil fuel has released greenhouse gases (GHG), such as CH_4 , SO_2 , NO_x , and other toxic gases or pollutants, which will cause global warming and acid rains (*Teh et al*, 2021). Biomass is also considered one of the main energy sources for both developed and developing countries. The energy demand had increased due to the increment in population. Malaysia is endowed with a lot of supplies of biomass resources such as plantation residue and agricultural residue. With the help of biomass resources, the combustion will not release greenhouse gases as

much as the conventional fuel. (Shafie et al, 2012) During the combustion process of biomass and waste, chlorides and sulphate containing melts may form on the surface of the superheater. The presence of chlorides and sulphate will introduce high-temperature corrosion after fine fly ash particles condense on the heat exchange surface. As the temperature becomes higher, the corrosion rate of the metallic components will be increased (Mudgal et al, 2014).

2.2.1 Biomass Plant in Malaysia

The literature review of *Nor Adilla Rashidi* shows that the energy consumption during the last few years in Malaysia had an increment. Malaysia is a country that rich in natural resources and it is blessed with a tropical and humid climate all year round which is a magnificent opportunity for fully exploiting agriculture and tropical forests' potential (Rashidi, 2022). Oil palm is the main source of biomass in Malaysia. Combustion involving oil palm had a few benefits including low sulphur content, non-toxicity, and high biodegradability, as well as good combustion efficiency stated by *Yusuf* and *Nomanbhay*. Due to the disadvantage brought by fossil fuels, the researcher had gained interest in using biomass energy as the main source for combustion in biomass plants in Malaysia. A study by *Chala (IEEE)* had shown that Malaysia's biomass consumption as fuel accounts for 16%, of which 51% of the biomass source is from palm oil and 22% from wood waste, and the other resources are obtained from other agricultural sectors and the agro-food industry. Griffin et al stated that direct combustion of biomass has generated 225MW of biomass electricity generation in Malaysia.

2.2.2 Combustion in Biomass Plant

Based on a handbook written by *Babcock and Wilcox*, there are three main types of boilers which are grate fired, bubbling bed, and circulating fluidized bed units. Usually, biomass fuel is burned in these three boilers. Combustion of biomass fuel in these boilers can generate electricity and a combination of heat and power (Sharp, 2010). The exothermal reaction between fuel and oxygen, which mostly produces carbon dioxide and water vapour, is known as combustion. Based on well-established technology, direct combustion is currently the main way to produce power globally. Modern combined heat and power (CHP) plants can reach an overall plant efficiency of about 90% when producing heat for district heating in addition to electricity (Dong Energy., 2012; Mandø., 2013). During high-temperature combustion of biomass fuel, the flue gases produced is corrosive because flue gases contain high concentration of alkali , sulphur and chlorine gases and particles. Presence of corrosive gases such as HCl and Cl₂ released by the combustion of biomass cause corrosion in the superheater tubes. Corrosive gases also form deposits on the surface of the tubes and this will limit the rate of heat transfer to the steam (Sharp, 2010). To eliminate the problem of corrosion, there are a few ways to solve the problem. Material selection and coatings are some of the ways to reduce the tendency of corrosion.

2.3 Corrosion issue in Biomass Plant

In this chapter, the corrosion issue will be discussed in detail. Corrosion is an important issue for superheater materials. The work-life of superheater materials will be reduced due to the corrosion. To increase the efficiency of the biomass plant, a high temperature is needed to burn the biomass fuel. Combustion of biomass fuel will release corrosive gases and cause corrosion on superheater tubes. There is 2 factor that will

cause the degradation of the superheater boiler system which is hot corrosion and alkali molten salt attack (Rujisomnapa et al., 2010).

2.3.1 Hot corrosion

Hot corrosion is a phenomenon that represents the high-temperature corrosion that takes place when molten salt is present on the surface of the metal (Landolt, 2007). There are two types of hot corrosion which are high-temperature hot corrosion (type I) and low-temperature hot corrosion (type II). At high temperatures (800 °C–950 °C), when the salt deposit on the metal or alloy is in a liquid form, Type I corrosion typically results through alloy-induced acidic fluxing or sulphidation. While type II hot corrosion often happens at low temperatures (600 °C to 750 °C) and is driven on by gas-induced acid fluxing or sulphidation. The temperature is lower than the melting temperature of the deposited salt. (Bradshaw et al., 2013; Khanna., 2012)

A large and growing body of literature has investigated the effect of high-temperature corrosion of Stainless Steel 304. One study by Ahmed in 2016 examined the effect of exposing specimens of 304 stainless steel to 0.5, 1.0, 1.5, and 2.0 M chloride concentrations and 70, 80, and 90 °C. The result shows that cracking starts to form at elevated temperatures and high concentrations of chloride, Besides, he also suggested when stainless steel 304 is applied at high temperatures and chloride concentration needs to take care of. In addition, the critical conditions of failure that occur by stress corrosion cracking must be identified (Ahmed, 2016).

2.3.2 Alkali Molten Salt Attack

Biomass-fired power generation often uses low-grade fuels having high concentrations of S, Cl, K, and Na in the process of producing thermal energy. Volatile alkali sulphates and chloride vapours easily interact with other ash components during

combustion, and these low melting point mixes are condensed to create molten deposits. The corrosion rate of the steel quickened with the rise in temperature, and the molten deposits may cause rapid material degradation owing to oxidation, sulfidation, chlorination, and even hot corrosion. The biomass-fired boiler's heat tubes are primarily corroded by molten alkali deposits (He et al., 2016). This section presents a review of recent literature review on alkali molten salt attacks. Based on the research of He et al (2016), Compared to mixes of NaCl-KCl and Na₂SO₄-K₂SO₄, the main corrosion effect factors in high-temperature corrosion are molten chlorine salts. Molten chlorides cause more serious fractures and fissures in the corrosion layer than molten sulphates because they are actively oxidising the environment.

From the research work of *Sofia* (2011), in a 5 percent O₂ + 40 percent H₂O atmosphere, the addition of KCl and NaCl significantly speeds up the corrosion of 304L stainless steel at high temperatures. When alkali reacts with the protective oxide, it produces alkali chromate, which starts the corrosion (Sofia, 2011). *Enestam* (2013) proved that at temperatures well below the salt or salt mixture's initial melting point, KCl and NaCl are both known to accelerate corrosion of low alloy and stainless steel superheater materials. When applied to steel surfaces at superheated temperatures, both NaCl and KCl exhibit high corrosivity. Both salts use vaporisation of metal chlorides and subsequent oxidation of the chlorides as the corrosion mechanism (Enestam, 2013).

2.4 Material for high-temperature application

Material selection is an important process for selecting the material to fulfill the requirement of the high-temperature application. In high-temperature applications, hot corrosion and alkali molten salt will reduce the work-life of the material. Therefore, the material selection process is important so that it can minimize and prevent the attack of corrosion selection that is resistant to corrosion under operating conditions.

2.4.1 Material used for high-temperature application

Currently, the temperature used for the combustion of biomass has risen to a certain range. Higher operation temperature caused severe problems to the lifespan of boiler and superheater material such as the hot corrosion. Hot corrosion will strongly affect the lifespan of the material. Based on the research work of Pierce, most of the material is designed for coating on the surface of the substrate to form a protective oxide layer to act as a diffusion barrier to oxygen and corrosive species (Pierce et al., 2019).

Conventional refractory metals such as niobium, tungsten, molybdenum, and tantalum have high melting points and good strength at high temperatures. Refractory metals can form oxide and sulphides with greater thermodynamic stability than corresponding chromium products. The corrosion rate of refractory metals and alloys is low even in a complex environment. However, the corrosion resistance of existing structural alloys can be improved by using refractory metals such as alloying element additions (Savolain, 2005).

Luo et.al (2012) conducted high-temperature corrosion behaviors of the superheater materials such as 20#steel, TP347H, and superalloy C22. The researcher investigated the corrosion behaviors of these superheater materials in the condition of synthetic salt-containing 80 wt-%KCl+20 wt-%K₂SO₄ under the oxidizing atmosphere at a temperature of 650°C for 218 hours. High content amount of Ni and Cr has improve the corrosion resistance of superheater materials. Thereby the superalloy C22 has the highest resistance , followed by TP347H and 20#steel. They also found that the corrosion degree is directly related to the mass loss (Luo et al., 2012).

In 2009, *Yin* and *Wu* (2009) conducted a research related to corrosion behavior of TP316L of superheater in biomass boiler with simulated atmosphere and deposit. In

their research, they conducted TP316L in an oxidizing atmosphere containing 0.01% HCl in the temperature range of 500-600°C. Compact FeSO₄ scales were formed on the surface of substrate. The function of the FeSO₄ scales is to act as a protective layer for corrosion (Yin and Wu, 2009).

Based on Mudgal's study, Midhani grade Superni 718 and Superni 600, a nickel superalloy, and Superco 605, a cobalt-based superalloy, have been chosen as the substrate materials. The high-temperature corrosion behaviour of superalloys was studied by the researcher in a actual waste incinerator scenario. The outcome demonstrates that there is an ash deposit on the substrate's surface. The Ni-based alloys Superni 718 and Superni 600 outperformed Superco 605 in terms of corrosion resistance among the three alloys when tested in an actual incineration environment.. Superni 718 and Siperni 600 have a better corrosion resistance due to the formation of protective oxide layer NiCr₂O₄ which provides more corrosion resistance to the surface of the substrate (Mudgal, 2016).

2.4.2 Properties of Molybdenum

Molybdenum is a naturally occurring elements and it is categorised as a "transition element" or "transition metal" since it is an element in the d-block of the Periodic Table of Elements, which contains groups 3–12. Molybdenum is a member of the class of "refractory metals" as well. These metals fall into a category that is highly heat- and wear-resistant. Molybdenum (Mo) is a common element that acts as a coating element to improve the thermal and wear resistance of substrate materials. Electrodeposition of molybdenum alloy produces a hard and protective surface that prevents corrosion and its components are not toxic (Rastogi, 2010). The important feature of molybdenum acts as an alloy in stainless steel and in alloy steel.

Molybdenum possesses a number of great properties. High melting temperature (2625 °C), high elastic modulus (330 GPa), high thermal conductivity (138 W mK), low coefficient of expansion (5.35 $\mu\text{m}^\circ\text{C}$ at 20 °C), low specific heat, high electrical conductivity, high strength at high temperatures, and high corrosion resistance are one of these properties (Hasan et al., 2018).

Molybdenum have an appearance of silvery-white. Molybdenum is advantageous in applications that require intense heat changes because it can resist severe temperatures without considerably expanding or softening, such as the production of aircraft parts, electrical connections, industrial motors, and filaments. Molybdenum can be used as an alloying component or as a flame-resistant coating for other metal (Rastogi, 2010).

According to Mesquita (2012)'s research, molybdenum addition had a significant favourable impact on the ability of industrial and laboratory lean duplex stainless steels to resist pitting corrosion in all investigated chloride-rich solutions. Due to its capacity to produce a more uniform grain structure and distribution of chromium in the lattice, the addition of molybdenum significantly increases this basic resistance by lowering the quantity of pitting sites that are present and therefore increased the efficiency of the passive protective film (Mesquita, 2012).

2.5 Electroplating

Electroplating is one of the methods that can be used to coat a layer on the surface of the substrate via current. Electroplating is a surface coatings protection technology that involves a chemical response of the constituents in an aqueous mixture subjected to galvanic excitation, resulting in the production of thin-film layers, or coatings, on the substrate of the material. Electroplating is also one of the few surface

finishing processes that can be used for a variety of functional and aesthetic goals. Besides, electroplating is an electrochemical plating technique that improves the visual appearance of materials while also increasing their service life and performance. The electroplating technique involves using an electrochemical process to apply a metallic coating on a metal substrate or the surface of a conducting substance. Electroplating has a number of advantages over other processes, including control accuracy, low energy requirements, flexibility, high production rate, economy in scaling, even coating, waste reduction, and the ability to coat more complicated topology and geometry. (Schlesinger et al., 2010).

2.5.1 Theory of electrodeposition

A number of techniques have been developed to form a coating on the surface of the substrate. One of the techniques is electrodeposition. Based on ASTM Int. B374-96, the definition of electrodeposition is the process of depositing a substance upon an electrode by electrolysis. Electrodeposition is a film-growth process that involves the electrochemical reduction of metal ions from an electrolyte, resulting in the development of a metallic coating on a base material. The electrodeposition process involves immersing the object to be coated in a vessel containing the electrolyte and a counter electrode, then connecting the two electrodes to an external power supply to allow current flow. The negative terminal of the power supply is connected to the object to be coated, reducing metal ions to metal atoms, which finally form the deposit on the surface (Gamburg, 2011). Compared to thermal spraying or CVD, electrodeposition is a more delicate coating process since there are a lot more factors that can influence how successfully the film is deposited as well as the electrochemical reactions that take place in the cell.

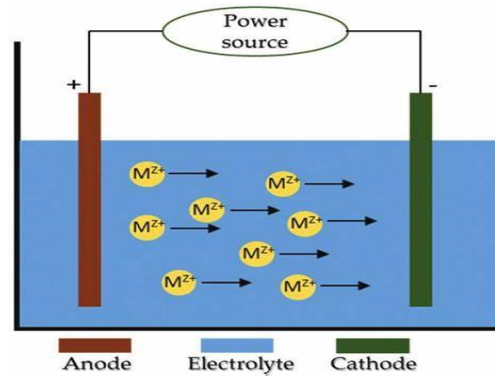
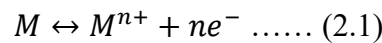


Figure 2.2 Schematic diagram of the electroplating process

The ability of a metal to be electrodeposited on the surface of a substrate is determined by its thermodynamic activity when in solution. A metal electrode is placed in an electrolyte that contains metal ions, and this builds an equilibrium.:



The capacity for a metal to either dissolve into solution and produce ions or, in the alternative, for ions to deposit as metal, is described by Equation 2.1. Thermodynamic equilibrium occurs when the net rate of these processes is zero. The Nernst equation describes the equilibrium potential:

$$E_{eq} = E^{\circ} + \frac{RT}{nF} \ln[M^{n+}] \dots\dots(2.2)$$

In order to deposit metal ions on the electrode, the potential must be shifted into the cathodic direction. Negative potential which also known as overpotential was needed to drive the reduction in this shift, η :

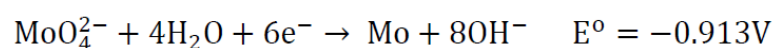
$$\eta = E - E_{eq} \dots\dots(2.3)$$

The energy needed to cause the metal ion to move freely across the electrolyte and have reduce reaction on the cathode is known as the overpotential. The overpotential required for metal ion reduction at a cathode is a unique value for every metal, as well as a variety of other factors like electrolyte pH, temperature, and stirring

velocity. The higher the overpotential of metal, the easier to have reduction reaction (Daniel, 2013).

2.5.2 Reduction of Molybdenum

In this research, molybdenum is selected to be deposited on the surface of the substrate. Molybdenum have a high-overpotential value The stable oxidation state of molybdenum is Mo (VI). Reducing Mo (VI) leads to a number of oxidation states, ranging from -2 to $+6$, with highly stable oxides of MoO_2 , MoO_3 (Daniel, 2013). When molybdenum oxide react with water, the product generated is anion molybdate, MoO_4^{2-} is formed , and its full metal reduction in water is given by:



The standard potential $E^0 = -0.913\text{V}$ is extremely high for the complete reduction of Mo(VI) to Mo. The electrodeposition of Mo element from aqueous electrolyte is due to hydrogen evolution reaction and oxyphilic nature of molybdenum. Hydrogen evolution reaction generates a large amount of gas bubbles and prevent the fresh electrolyte to react with substrate (Siti, 2018). At pH 6.8, Mo has a low hydrogen overpotential ($E_{\text{eq}} = -0.430\text{ V}$), which favourable thermodynamic process than metal ion reduction. As a result, the hydrogen evolution reaction (HER) occurs frequently and uses up a lot of the applied current. The electrodeposition of metallic molybdenum usually produced poor results, with low current efficiency. The high reducing overpotentials of molybdenum results in brittle coating (Syed, 2015).

2.6 Coating

In order to produce materials, pieces of machinery, and machine parts with desirable surface properties like corrosion, erosion, and wear resistance at a reasonable

cost, surface coating is often used. Surface coatings can help improve corrosion, erosion, impact breakage, and abrasive wear resistance. Surface engineering is a branch of science concerned with methods for accomplishing desirable surface requirements as well as their behaviour in the service of engineering components. The effect of surfacing on component life and performance is determined by the surface material, alloy, service circumstances, and application method (Shibe et al., 2013)

The coatings need to have a high level of adhesion to the substrate, a low level of coating discontinuity (porosity), and a high resistance to the flow of electrons in order to have strong corrosion resistance. The coating's thickness is another criterion after that. The coating's resistance increases with coating thickness. The coating's diffusion rate for ions like Cl^- and for H_2O must be low (Ahmad, 2016).

There is a lot of coating method available due to the requirement of the application. Different coating methods provided different outcomes in the form of microstructure, effectiveness, suitability, and durability. However, the most important function of the coating is to prevent corrosion and wear. The corrosion process causes a deterioration in the mechanical characteristics of the materials while the corrosion products are discharged in various forms that may result in a more harsh corrosive environment or negative side effects in certain applications. For the desired application, several coating techniques have diverse deposition mechanisms, all with advantages and disadvantages (Fotovvati et al., 2019).



Figure 2.3 Schematic diagram for Cross-section of the molybdenum coating on stainless steel 304 substrate

2.6.1 Coating Techniques

In general, only a few of the coating methods are applicable and effective such as physical vapor deposition (PVD), chemical vapor deposition (CVD), micro-arc oxidation (MAO), sol-gel, thermal spray, and polymer coatings. Each technique has a variety of applications because to its various deposition techniques, materials, second phases, thicknesses, and densities. In the following part, those coating methods will be discussed briefly.

Physical vapor deposition (PVD) is a coating process that is well known for its excellent corrosion and wear resistance. In general, it is applied on the surface of the material that is exposed to harsh and corrosive environments in different fields and the coatings are also applied as decorative objects to industrial parts. Generally, PVD is used to deposit thin coatings on a variety of workpiece surfaces by the evaporation or momentum collision of the source material. Evaporation is a process used to move atoms from a source to a substrate at a specific distance. Thermal, electron-beam, or pulsed laser techniques are used to heat the materials to be deposited until they reach their sublimation temperature. Sputtering and evaporation are the two most typical PVD techniques. In simple words, PVD is a high vacuum process that converts solid/liquid materials to a vapour phase, followed by metal vapour condensation, which results in the formation of a solid and dense film. In physical vapor deposition, there is no chemical reaction occurs (Rane et al., 2018).

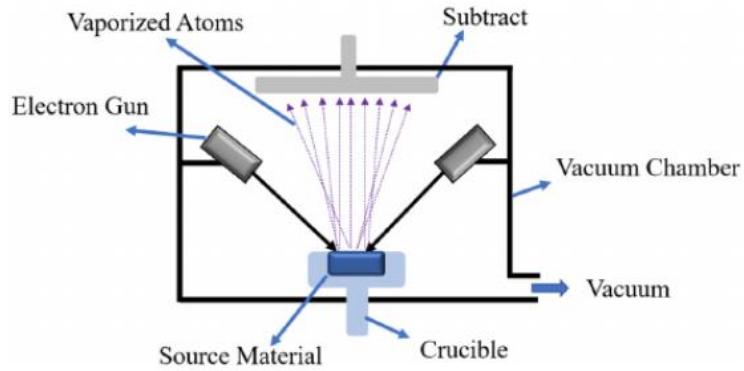


Figure 2.4 Schematic view of a physical vapor deposition (PVD) machine using electron beam as the heat source (Fotovvati et al., 2019)

There are other types of vapor deposition known as chemical vapor deposition (CVD). A precursor gas or gases are injected into a chamber containing one or more heated objects to be coated in order to create a coating by chemical vapour deposition. A thin coating is deposited on the surface as a result of chemical reactions that take place close to heated surfaces. Along with it, unreacted precursor gases and chemical by-products are produced and ejected from the chamber (Creighton and Ho, 2001). Unlike PVD, which involves a mixture of gases and the bulk surface of the material, CVD involves a real chemical interaction that results in the chemical decomposition of some of the specific gas constituents and the formation of a solid coating on the surface of the base material (Behera et al., 2020) .

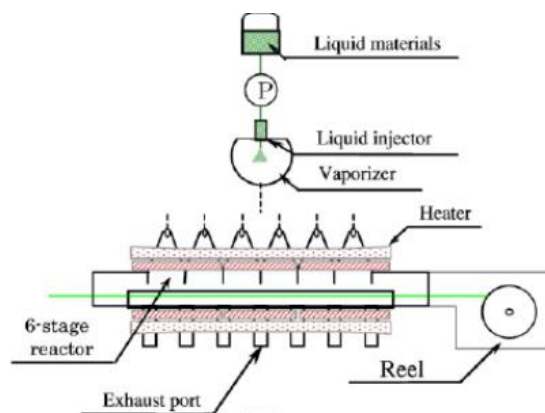


Figure 2.5 Schematic chemical vapor deposition (CVD) setup, mechanical parts, and operation mechanism (Fotovvati et al., 2019)

Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD) are promising processes for producing coatings composed of molybdenum and molybdenum alloys. Most materials can be deposited using these procedures, regardless of their melting point or degree of reactivity. High operating temperatures and pressures are necessary for CVD, which may have an impact on the type of material and size of substrate.

Thermal spray coating refers to a group of process where a set of designed materials are melted using a plasma, electric, or chemical combustion heat source, and the melted materials are then sprayed onto the surface to create a protective layer. These sorts of corrosion- and wear-resistant coatings are dependable materials. In this technique, the components are heated to a molten or semi-solid phase and sprayed on the substrate with a high-speed jet. The heat source used to do this is typically chemical combustion or plasma discharge. Thermal spray coating techniques may reach thicknesses of up to several millimetres, which is a significant improvement above the thicknesses afforded by PVD, CVD, or electroplating procedures. Additionally, a reasonably large surface area of a substrate can be easily covered by a thermal spray coating using a variety of materials, including refractory metals and metallic alloys, ceramics, polymers, and composites. Based on their properties and process requirements, thermal spray coatings are divided into various categories. Plasma, detonation, warm/cold, high-velocity air fuel (HVOF), high-velocity oxyfuel (HVOF), flame, and wire arc spraying are the most popular categories. (Fotovvati et al., 2019)

One of the often used coating techniques in the oil and gas industry is thermal spray. *Riastuti* had successfully created molybdenum coatings on the surface of stainless steel 316L using the thermal spraying method according to his research. The molybdenum coating on stainless steel 316L can function as a barrier that can be