UNIVERSITI TEKNIKAL MALAYSIA MELAKA (UTeM)

STUDY OF CORROSION RESISTANCE AND WEAR BEHAVIOUROF SOL GEL COATING ON AA7075

Report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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ABSTRAK

Tujuan kajian ini yang mengetahui tingkah laku kehausan dan rintangan kakisan lapisan sol gel pada aloi aluminium 7075.AA7075 digunakan secara meluas untuk pembuatan pesawat kerana ketumpatan yang rendah kepada nisbah berat badan dan kekuatan yang tinggi .Memandangkan aluminium ini beroperasi dalam persekitaran yang agresif dalam udara atmosfera , aluminium ini mestilah bersalut untuk mengelakkan corrosion.Walau bagaimanapun , wujud kebimbangan teknik biasa tentang teknik kromat yang biasa digunakan untuk melindungi aloi aluminium kerana ia adalah berbahaya dan malahan boleh menyebabkan kanser. Salutan sol-gel menjadi subjek kajian untuk melindungi AA7075 kerana secara kimianya bersifat lengai dan telah terbukti dalam kajian terdahulu mempunyai sifat tahan karat yang baik. Analisa terhadap sampel yang disalut dengan teknik sol gel mengesahkan kewujudan silica sebagai lapisan salutan. Salutan sol gel menunjukkan perlindungan terhadap kakisan pada manakala keputusan daripada ujian kehausan menunjukkan satu trend prestasi apabila kepekatan TEOS berubah.

ABSTRACT

The purpose of this research is to study the wear behaviour and corrosion resistance of sol-gel coating on aluminium alloy 7075. AA7075 is widely used for aircraft manufacturing due to its low density to weight ratio and high strength. As this aluminium operates in aggressive environments in atmospheric air, the aluminium must be coated to prevent corrosion. However, the commonly used chromating technique become concern because it is hazardous and even carcinogenic. The sol gel coating become subject of interest to protect the AA7075 due to its chemical inertness and have been proven for excellent chemical resistance. The coated sample analysed by XRF confirmed the presence of silica as the coating layer. Samples observed under SEM shows the presence of crack on the coatings. The sol gel coating shows corrosion protection at certain TEOS concentration whereas the results from wear study shows a pattern of performance as the TEOS concentration changes.

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LIST ABBREVIATIONS, SYMBOLS, NOMENCLATURE

Al	=	Aluminium
C	=	Carbom
Cl	=	Chlorine
H	=	Hydrogen
ml	=	Mililiter
Si	=	Silica
HCl	=	Hydrochloric acid
TEOS	=	Tetraethyl orthosilicate
MAPTS	=	Methacryloxypropyl trimethoxysilan
Zr	=	Zirconia
μ	=	Micrometer

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Aluminium alloy 7075 (AA7075) is an aluminium alloy, with zinc as the primary alloying element. It is widely used in manufacturing parts for transportation industries especially for aircraft. While this metal alloy is useful because of their physical characteristics such as good fatigue strength and high strength to weight ratios, it is highly susceptible to corrosion in aggressive environments. Nowadays, there are several methods to protect the aluminium and its alloys from corrosion such as coatings (metallic, Inorganic, conversion and organic coatings), control of environment (operating variable i.e. pH, dissolved oxygen, temperature and etc.) and corrosion inhibitors (organic and inorganic additives).The most common technique to protect most metal alloy, is chromium conversion coating (CCC). This technique provides excellent corrosion protection and good adhesion properties. However, the use of chromates in the coating has generated serious hazardous problems since the active ingredient in this coating, hexavalent chromium is strongly carcinogenic.

In response, one of the coatings method- sol-gel coating- has been discussed as one of the potential alternative towards the hazardous chromium conversion coating. Compared to CCC, sol-gel coating is environmental friendly and “green” technology. Basically, sol-gel is a process where oxide network is created by progressive condensation reactions of molecular precursors in a liquid medium. In sol-gel process, a system of colloidal particles in the solution (sol) become a macroscopic material (gel), which is merged by a liquid. As the liquid evaporates, a strong glass-like material remains. This is part where interest for sol-gel process lies because the deposited ceramic coating is chemically inert compared to CCC.

Even though prior researches and studies have proven excellence corrosion resistance of sol-gel coating on various metal alloy, but very little was found in the literature on the question of wear behaviour of this coating towards the substrate. This research aims to know the performance of sol-gel coating particularly on AA7075 in terms of wear behaviour, adhesion as well as corrosion resistance and how the variation of certain parameter and heat treatment process would affect those performance.

1.4 Problem Statement

There are many techniques to protect the aluminium but the most efficient technique that is widely used is chromium conversion coating. The process is carried out by oxidising the aluminium in a bath containing chromic acid and accelerating anions. The protective layer consists of thin film of chromate and chromium oxide. However, the hexavalent chromate compounds is well-known for its toxicity and carcinogenic. Sol –gel technology provides a unique and highly promising approach to tackle this challenge. But, it is important to know corrosion resistance and wear behaviour of the sol-gel coating because they determines the performance and durability of the coating. Any significant change in wear behaviour and corrosion properties by altering the concentration of the precursor used is observed in this research.

1.2 Objectives

1. To characterize the hybrid TEOS-MAPTS sol-gel coatings on the AA7075 substrate produced at various TEOS concentration
2. To determine the corrosion resistance of sol-gel coating on the AA7075 substrate at various TEOS concentration.
3. To identify the wear behaviour of sol-gel coating on the AA7075 substrate at at various TEOS concentration in 3.0 % NaCl solution.

1.3 Scope of Study

The focus of this study is to study the adhesion, wear behaviour and corrosion resistance of sol-gel coatings on aluminium alloy 7075 substrate. The sol-gel is prepared by using TEOS and MAPTS as precursors. Different sol is prepared by varying the TEOS concentration.. The precursors must be prepared first by few stages before can be proceed for coatings. The coating of aluminium alloy will be done at several TEOS concentrations to know the optimum deposition for good corrosion resistance and wear behaviour. Further, dip-coating method is used to coat the substrate with the sol prepared After completing the coating process, the characterisation the deposited coating will be done using Scanning electron microscopy(SEM) and X-ray fluorescence(XRF). The wear behaviour and corrosion resistance is determined using suitable testing .Effect of concentration of TEOS as precursor is compared in studying the corrosion and wear behaviour of the coating.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviews the previous researches and studies on the sol gel coating and related topics. Thorough the literature review, the principle of sol gel process its performance as protective coating have been studied and discussed.

2.2 Sol-gel

2.2.1 Mechanisms

Sol gel is a chemical synthesis technique for preparing glasses, gels and ceramic powder. Sol-gel synthesis may be used to prepare materials with a variety of shapes, such as porous structures, thin fibers, dense powders and thin films as illustrated in Figure 2.1.

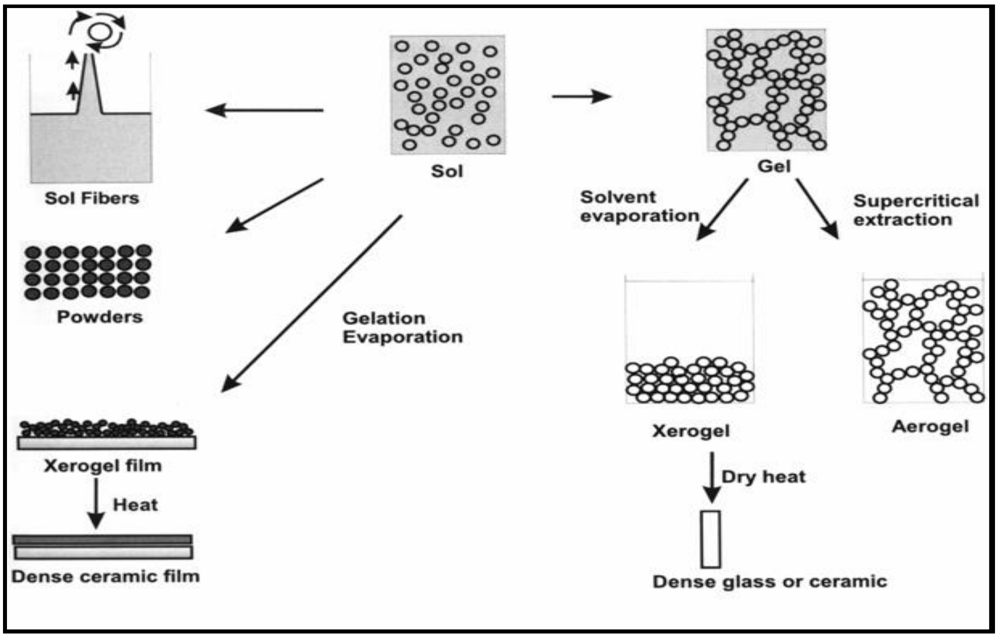

Figure 2.1: Sol-gel synthesis to produce various materials.

(Source: https://str.llnl.gov/str/May05/Satcher.html)

Brinker and Scherrer (1990) defined sol gel process as creation an oxide network by progressive condensation reactions of molecular precursors in a liquid medium. Precursor is a compound that involved in chemical reaction to yield another compound. TEOS , a colourless liquid with chemical formula $Si(OC_2H_5)_4$, is commonly used as precursor in sol gel route due to its property which is easy to convert to silicon dioxide.

Generally, the sol gel reaction starts with hydrolysis of precursors then followed by condensation reaction to produce a macroscopic gel as illustrated in Figure 2.2.

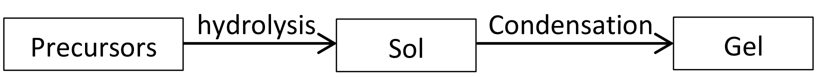

Figure 2.2: Basic sol-gel reaction

There are two ways to prepare sol gel coating- the inorganic method and organic method. The inorganic method involves the two evolution of networks through the formation of colloidal suspension and gelation of the sol whereas for organic method, it generally starts with a solution of metalloid alkoxide precursors $M(OR)_n$ in an alcohol or other low-molecular weight solvent. M here denote a network-forming element, such as Zr, Ti, Al, Si, etc.; and R is typically an alkyl group ($C_x H_{2x+1}$). (Wang and Bierwagen, 2009).

2.2.2 Sol-gel as Protective Coating

Metal oxide coatings such TiO_2, ZrO_2 and SiO_2 have a very good chemical stability and can provide good protection to metal substrate. .SiO_2 improves the oxidation and acidic corrosion resistance of metals under different temperatures due to its high heat resistant and chemical resistance (Galliano, 1998).

Silica coating has been used on surfaces of steel, carbon and aluminium to promote adhesion, minimise photodegradation, or prevent material oxidation and corrosion (Schwarz, 1999).The silica coating by the sol-gel process is conducted in an organic solvent. The precursor of TEOS hydrolyse in the presence of water and catalyst, followed by condensation with surface metal hydroxyls. The Si-O-M chemical linkage is established between surface metal atoms (M) and TEOS, followed by lateral polymerization and finally the formation of a 3-D network via the formation of siloxane bonds with increasing TEOS concentration and hydrolysis.

Prior researches have stressed out the advantages of using sol-gel method to coat the metal alloy. Sol-gel coating is environmental friendly. Osborne et al.,(2001) and Vignesh et al., (1998) pointed that sol gel coating is a promising approach to commonly used chromating technique to protect the aluminium alloy. The hexavalent chromate compound in chromating technique is toxic and carcinogenic. The use of sol-gel technique is a good alternative to overcome this problem. According to Hu et al., (2012), the sol gel coating are formed by "green" technologies. Compounds used do not introduce impurities into the end product as initial substances. Meanwhile, Alain (1998) stated that the association of the solid colloidal state with a liquid medium avoid any pollution by the eventual dispersion of dust.

In sol-gel process, inorganic solids can be produced and processed at temperatures which are considerably lower than those in conventional methods (Wright and Sommerdijk, 2001).

Sol-gel coating can be applied to produce multilayer coatings. Further, this method also allow the preparation of composite which cannot be obtained by other methods, such as organic-inorganic hybrid materials (Wright and Sommerdijk, 2001).

However, in spite of the advantages of sol-gel oxide coating offers, previous researches have pointed few weaknesses of inorganic oxide coating. Wang and Bierwagen(2009) stated that the inorganic oxide films are brittle and it is difficult to achieve thicker coatings without cracking. They found that relatively high temperature is always needed to achieve good properties of inorganic oxide coating.

Rahimi et al., (2013) suggested that the formation of inorganic-sol gel derived films may not provide adequate barrier properties to prevent the corrosion, due to pores and crack present after the drying procedure.. While Conde et al.,(2003) reported sol-gel materials based solely on inorganic systems usually quite porous, unless densification is carried out at high temperature, which can disturb the properties of base metal.

2.2.3 Hybrid sol gel coating

The hybrid sol-gel coating is introduced which combine the properties of inorganic and organic materials. The development of hybrid sol-gel coating solve the temperature problem as low curing temperature is needed which preserve the integrity of metallic substrate and at the same time reduce the risk of cracks appearance (Rahimi et al.,2013). Organic-inorganic hybrid coatings can easily form a thicker coat in micrometer scales without cracks, which can improve the protective ability of sol-gel coatings The inorganic coating is achieved by combining different precursors. Chou et al., (2001) in their experiment by using TEOS and MPTS on 304 stainless steel found that the hybrid coating deposited is relatively dense, crack free and uniform. Rahimi et al.,(2013) in their research of coating AA5053 through sol-gel technique showed that crack-free films with smooth surface is obtained by mixing TEOS and GPTMS as precursors.

The microstructure of the silica coating obtained through sol-gel process depends on the hydrolysis and condensation reactions which are controlled by few factors. One of the factors is concentration of TEOS. According to Barbé et al,(2005), an increase in the TEOS concentration contributes to an increase in the solution's viscosity and thus increase the rate of hydrolysis and condensation reaction.

2.2.4 Dip coating

According to Brinker et al.,(1991) dip coating is a process of withdrawing the substrate from the a fluid sol(gravitational draining and solvent evaporation) and followed by further condensation reactions, which shrink the film into a gel. Once it has collapsed fully, a dense sol-gel thin film remains. During the deposition stage there is a constant competition between condensation reactions and evaporation. The condensation reactions act to stiffen the structure while the evaporation compacts it. The entire process, including the aggregation, gelation and drying occurs within seconds for the dipped slides (Mellor, 2001).Figure 2.3 illustrates the dipping process. The substrate is usually withdrawn vertically at a speed U_0. As the solvent is evaporating and draining, the fluid film stops at drying line x=0.The process is steady state when the receding drying line velocity equals U_0.

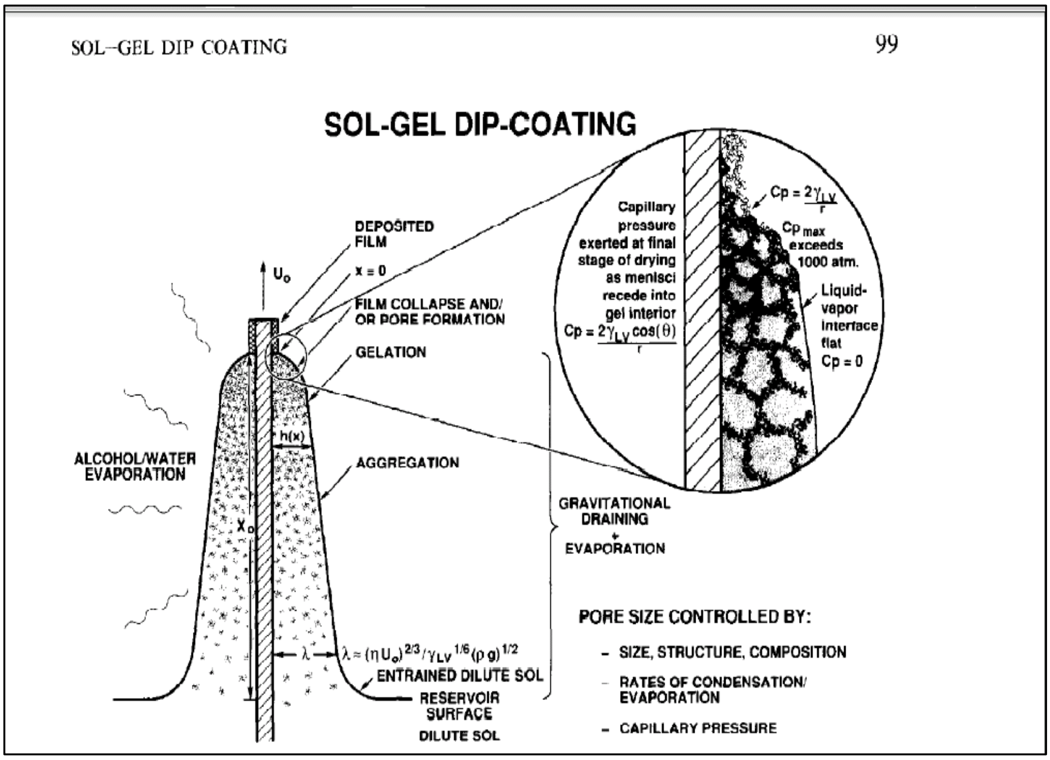

Figure 2.3: Dip-coating mechanism

Source : Brinker et. al, (1991)

2.2.5 Applications

Nowadays, the application of sol gel of sol gel is widespread. Sol-gel technology is applied in development of optical materials, chemical sensors, as well as coating.

2.3 Aluminium

Aluminium is a silvery-white-look, soft, and ductile metal from Group 13.It is the third most abundant element after oxygen and silicon. Its low density property and ability to resist corrosion due to the passivation reaction make it a significant choice as structural materials for transportation. When aluminium surface is exposed to the atmosphere, the natural oxide film is formed which are responsible for its corrosion resistance. However, this natural oxide film cannot protect the metal in severe corrosive environments (Vignesh et al., 2013).

2.3.1 Aluminium alloy

Aluminium always alloyed with other elements to improve its mechanical properties because pure aluminium is too soft for engineering applications. Among the typical alloying elements for aluminium is silicon, copper, zinc , manganese and magnesium.

Aluminium alloy can be classified into two categories which are casting alloy and wrought alloy.Casting alloy have to be remelted an then casted. They are delivered in the form of T-bars,which can be very easily remelted.The major alloy elements include 2xx series(AL-Cu), 3xx series(Al-Si+ Mg and/or Cu) and 5xx series(Al-Mg).

Most aluminium is used for wrought product. The designation “wrought” means that certain aluminium alloys are available primarily in the form of worked products such as plate, foil, sheet, rod and extrusions. They can divided into two classes: non-heat treatable and heat treatable alloy. Non-heat treatable alloys is developed by solid solution hardening and strain hardening from the annealed temper. The properties depends on major impurities. The non-heat treatable alloy are in the 1xxx, 3xxx, 4xxx and 5xxx series. On the other hand, heat treatable wrought alloy contain one or more of the elements of copper, zinc, magnesium and silicon that are soluble in aluminium in considerable amounts at elevated temperature, but t a much smaller degree at room temperature (John, 1984).

2.3.2 Aluminium alloy 7075

Aluminium alloy 7075 is an aluminium alloy with zinc as its primary alloying elements. The presence of zinc as major alloying element will increase the strength and allow hot or cold precipitation hardening. The composition of elements present in AA7075 is shown in Table 2.1. AA7075 is widely used in transport applications especially in aeronautical industries due to their attractive comprehensive properties such as low density, high strength and resistance to fatigue. Table 2.2 shows the mechanical properties of AA7075.

Table 2.1: Composition of alloying elements in AA7075

Elements	Content wt. (%)
Aluminium	90
Zinc	5.6
Magnesium	2.6
Copper	1.6
Chromium	0.23

Table 2.2: Mechanical properties of AA7075

Density(kg/m^3)	2810
Fatigue strength(MPa)	159
Fracture toughness($MPa\text{-}m^{1/2}$)	25
Ultimate Tensile Strength(MPa)	572
Elongation at break(%)	11
Modulus of elasticity(GPa)	71.7

2.4 Wear

2.4.1 Definition

One of the important properties in studying the mechanical performance of a coating is wear behaviour. According to Bayer (2004), wear is a loss of material from the interface of two bodies when subjected to relative motion under load. Wear can also be defined as a process where interaction between two surfaces solids within the working environment results in dimensional loss of one solid, with or without any actual decoupling and loss of material. The scope of the working environment which affect wear is not only features and loads such as reciprocating, unidirectional sliding, rolling, and impact loads, speed, temperature, but also include different types of counter-bodies such as gas, liquid and solid. Compared to corrosion, wear is always, even not necessarily, referred to mechanical action whereas corrosion is due to chemical action.