EFFECTS OF HYDROXYAPATITE COATING WITH OXIDE INTERLAYER ON BIOACTIVITY PERFORMANCES IN CoCrMo ALLOY

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ABSTRACT

Cobalt-chromium-molybdenum (Co-Cr-Mo) alloys have been reported difficult to bond directly on hard tissues owing to encapsulation by fibrous tissues. Several attempts have been made to improve the situation including coating with a bioactive layer which is mainly hydroxyapatite (HA). Various HA coating methods have been introduced but massive micro crack surface, delamination and low adhesion strength of HA coating are still the major concerns that cause the harmful release of metal ions. In this study, an oxide interlayer on Co-Cr-Mo alloys was developed through thermal oxidation prior to HA coating with the objective to provide better anchorage of HA coatings on the substrate surface, reduce metal ions release and at the same time enhancing the cell attachment. The thermal oxidation process was conducted in a muffle furnace at different temperatures (850°C, 1050°C and 1250°C) for 3 hours to create an oxide interlayer on the substrate surface. It was followed by coating HA on the bare material and on the oxidized substrates using sol gel dip coating technique. Scratch test results showed that the bonding strength of the HA on the oxide interlayer is markedly higher than the HA coated substrates without oxide interlayer. It seems that rough surface of oxide interlayer provides better mechanical interlocking of HA particles to the substrate surface. Inductively coupled plasma-mass spectrometry (ICP-MS) test illustrated that the release of Co and Cr ions from the HA coated oxidized substrates reduced significantly after 28 days immersion as compared to bare material and HA coated substrates without oxide interlayer. This indicates that oxide interlayer is able to act as an additional barrier to suppress the metal ions release. Similarly, the HA coated substrates with oxide interlayer demonstrate strong attachment and proliferation of cells than the HA coated substrates without oxide interlayer. It is concluded that the introduction of an intermediate oxide layer on Co-Cr-Mo substrate prior to HA coating has shown a positive effect in terms of increment of the adhesion strength of HA coating as well as cell bioactivity performance.

ABSTRAK

Kobalt-kromium-molibdenum (Co-Cr-Mo) aloi dilaporkan sukar untuk mewujudkan ikatan secara langsung dengan tisu tulang kerana pengkapsulan oleh tisu bergentian. Beberapa usaha telah dibuat untuk memperbaiki situasi ini termasuklah dengan menyalut lapisan bioaktif terutamanya hidroksiapatit (HA). Pelbagai kaedah menyalut HA telah diperkenalkan namun permukaan retak mikro yang besar, pengelupasan dan kekuatan ikatan yang lemah dari salutan HA masih menjadi perhatian utama kerana ia menyebabkan pembebasan ion logam yang berbahaya. Dalam kajian ini, satu lapisan oksida di atas Co-Cr-Mo aloi telah dibangunkan melalui kaedah pengoksidaan terma sebelum salutan HA bertujuan untuk menyediakan ikatan yang lebih baik bagi salutan HA di atas permukaan sampel, mengurangkan pembebasan ion logam dan dalam masa yang sama meningkatkan pelekatan sel. Proses pengoksidaan terma dijalankan di dalam relau redup pada suhu yang berlainan (850°C, 1050°C dan 1250°C) selama 3 jam untuk membentuk lapisan oksida di atas permukaan sampel. Seterusnya ialah penyalutan HA di atas bahan asal dan sampel teroksida menggunakan teknik salutan bercelup. Keputusan ujian calar menunjukkan kekuatan ikatan HA di atas lapisan oksida lebih tinggi berbanding salutan HA di atas sampel tanpa lapisan oksida. Ia seolah-olah permukaan kasar lapisan oksida telah menyediakan pautan mekanikal yang lebih baik bagi zarah HA ke atas permukaan sampel. Ujian induktif berpadu plasma-besar spektrometri (ICP-MS) menggambarkan pembebasan Co dan Cr ion daripada salutan HA teroksida sampel menurun dengan ketara selepas 28 hari rendaman berbanding dengan bahan asal dan salutan HA sampel tanpa lapisan oksida. Ini menunjukkan bahawa lapisan oksida dapat bertindak sebagai halangan tambahan untuk menyekat pembebasan ion logam. Malah, salutan HA sampel dengan lapisan oksida juga menunjukkan lekatan yang kuat dan percambahan sel berbanding salutan HA sampel tanpa lapisan oksida. Kesimpulannya, pembentukan pengantara lapisan oksida di atas Co-Cr-Mo sampel sebelum salutan HA menunjukkan kesan positif dari segi peningkatan kekuatan ikatan salutan HA dan juga prestasi bioaktiviti sel.

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LIST OF ABBREVIATIONS

µg/l	-	Microgram per litre
µm	-	Micrometer
AAS	-	Atomic Absorption Spectrometry
AFM	-	Atomic Force Microscopy
AlN	-	Aluminum Nitride
CaP	-	Calcium Phosphate
CO_2	-	Carbon dioxide
Co-Cr-Mo	-	Cobalt-Chromium-Molybdenum
Cr_2O_3	-	Chromium Oxide
CrN	-	Chromium Nitride
CTE	-	Coefficient Thermal Expansion
DNA	-	Deoxyribonucleic Acid
EDX	-	Energy Dispersive X-ray Spectroscopy
FCC	-	Face Centered Cubic
FDA	-	Food and Drug Administration
FESEM	-	Field Emission Scanning Electron Microscopy
FHA	-	Fluoridated Hydroxyapatite
g	-	Gram
g/cm^3	-	Gram per cubic centimetre
GPa	-	Giga Pascal
HA	-	Hydroxyapatite
HCP	-	Hexagonal Close Packed
HDMEC	-	Human Dermal Microvascular Endothelial Cells
HPMEC	-	Human Pulmonary Microvascular Endothelial Cells
HUVEC	-	Human Umbilical Vein Endothelial Cells

ICP-MS	-	Inductively Coupled Plasma-Mass Spectroscopy
ISO	-	International Organization for Standardization
min	-	Minutes
ml	-	Millilitre
mm	-	Millimetre
mm/min	-	Millimetre per minute
MOM	-	Metal-On-Metal
MPa	-	Mega Pascal
MSCs	-	Multipotent Stromal Cells
N	-	Newton
N/min	-	Newton per minute
ng/ml	-	Nanogram per millilitre
nm	-	Nanometer
nmol/l	-	Nanomole per litre
PCL	-	Poly ε-caprolactone
PIII	-	Plasma Immersion Ion Implantation
PLD	-	Pulse Laser Deposition
ppb	-	Part per billion
ppm	-	Part per million
PVD	-	Physical Vapour deposition
Ra	-	Surface roughness
rpm	-	Rotation per minute
RPMI 1640	-	Roswell Park Memorial Institute 1640 medium
SBF	-	Simulated Body Fluid
SEM	-	Scanning Electron Microscopy
TCP	-	Tricalcium Phosphate
TiN	-	Titanium Nitride
TJR	-	Total Joint Replacement
TNF-α	-	Tumor Necrosis Factor
XRD	-	X-ray Diffraction Microscopy

LIST OF SYMBOLS

°C	-	Degree celcius
°C/min	-	Degree celcius per minute
Å	-	Armstrong (1 x 10^{-8}cm)
µ	-	Micro
~	-	Approximately
>	-	More than
<	-	Less than
wt.%	-	Weight percentage
E	-	Young's Modulus
σ_y	-	Yield Strength
σ_{UTS}	-	Tensile Strength
σ_{end}	-	Fatigue Limit

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter describes the general overview on the current issues such as corrosion resistance, release of toxic metal ions and bone bonding ability which is commonly encountered in biomaterial implants, followed by the recent work done to overcome these problems. Based on the literature study, the major problems were selected to be solved and become the problem statement for this research works. The determination of the problem statements, objectives, scopes, significance of the research and hypothesis of the results are also discussed in the following sections.

1.2 Research Background

Nowadays, the field of biomaterials is not new and has immense importance of the mankind as the existence and longevity for some of less fortunate human beings, for the aged population to increase their life span and also for patient who

experienced severe injury due to traumatic events. Apart from these reasons, young and dynamic people like athletes also need replacements due to fracture and excessive strain to help them continue normal life activities. The demands on biomaterial implants such as artificial joints, dental implants, bone plates, wires, and stents are continually increased especially after the world war and when global terrorism frequently strikes in today's challenging world environment (Afolaranmi *et al.*, 2011; Okur, 2009).

Currently, the uses of metallic biomaterials have widespread in replacing the damaged structural components of the human body due to their excellent mechanical properties such as high corrosion resistance, wear resistance, fatigue strengths and toughness. Some of their uses in medical devices are artificial joints, dental implants, bone plates, wires and stents. There are various types of biomaterials that have been used as implants such as steels, cobalt and titanium based alloys. Comparing these three alloys as metallic biomaterial in terms of biomedical properties and availability, cobalt based alloy which also known as Cobalt-Chromium-Molybdenum (Co-Cr-Mo) alloy are advantageous compared to stainless steel and titanium alloys. The biocompatibility of Co-Cr-Mo alloy is closely related to its excellent corrosion resistance, mainly due to the high chromium content (~ 30 wt.%) which is higher than 316L austenitic stainless steel (~ 18-20 wt.%) (Okur, 2009; Qingliang *et al.*, 2010; Santavirta *et al.*, 1998). Though stainless steels may have advantage in low cost, but because of its high toxicity of metal ions release and susceptibility in stress, they still have limited use in clinical practice.

Besides that, Co-Cr-Mo alloy also possess high fatigue strength in the porous-coated condition compared to titanium alloy, which is better potential use in extensively porous-coated hip systems, especially in the smaller size of implants that may be subjected to higher stresses (Disegi *et al.*, 1999; Giacchi *et al.*, 2011). Their favourable in tribological behaviour also make this alloy particularly suitable for metal-on-metal bearing surface such as the acetabular cup of total hip replacements (Igual Muñoz and Mischler, 2011). Furthermore, restrictions used of titanium alloy due to its inferior tribological properties and high costs when compared to stainless

steel and Co-Cr-Mo alloy, caused surgeon to shift for better biomaterial (Okur, 2009). In short, Co-Cr-Mo alloy is much preferable to be used as biomedical implant due to its combination of excellent mechanical properties, its workability to be forged into complex shape with compromise surface finish and low cost compared to other biomaterials alloy (Giacchi *et al.*, 2011; Lutz *et al.*, 2011). However, much effort is devoted to the design, synthesis and fabrication of Co-Cr-Mo orthopaedic implants in order to obtain long term stability and better anchoring between the metal implant and the bone. Essentially, this means the ability of the implant to sustain the dynamic and static loads when implanted in the human body. At the same time, the implant should also able to accelerate bone healing at the early implantation, with a very small failure rate and minimal discomfort for the patient. These factors are definitely influence the rate of implantation cost and restricting the widespread application of Co-Cr-Mo as biomedical implants. Since the formation of a living bone with direct contact to the implant surface is a critical issue and has received most attention from researchers, the common question arises as how to attain a better integration of the implant surface morphology.

Many attempts have been made to investigate and modify the implants surface in order to improve bone bonding ability. In recent years, there has been increasing interest to apply hydroxyapatite (HA) as coating layer since it is able to promote osseointegration on biomaterials implant (Sepehr *et al.*, 2013; Shaylin and George, 2012). HA coating can be applied by a number of methods such as pulsed laser deposition (PLD) (Rajesh *et al.*, 2011), plasma spray deposition (Cao and Liu, 2013; Coyle *et al.*, 2007), biomimetic precipitation (Shaylin and George, 2012), sol-gel dip coating (Hongjian and Jaebeom, 2011) and electrochemical deposition (Lu Ning and Jing Li, 2011).

Among these methods, sol–gel dip coating technique has been attracted much attentions, due to its many advantages, which include high product purity, homogeneous composition and low synthesis temperature and low processing cost (Mathews *et al.*, 2009). The low processing temperature and fusion of the apatite crystals have been the main attraction of the sol–gel dip coating process, in

comparison with high temperature process such as thermal spray. High temperature used in thermal spray has caused researchers to shift for alternative method that uses low processing temperature. In addition, sol–gel dip coating process also can produce mixture of fine grain microstructure from nano-to-submicron crystals. These crystals are reported to have good biocompatibility with host tissue (Hongjian and Jaebeom, 2011; Kim *et al.*, 2004) and able to enhance the cell adhesion, proliferation and growth at the interface of implant materials.

Therefore, the aim of this research was to evaluate the effects of oxide interlayer created through thermal oxidation process prior to HA coating on Co-Cr-Mo alloy. The purpose to create oxide interlayer on Co-Cr-Mo alloy is to provide better adhesion of HA coating due to many researchers claimed that direct coating can caused delamination and cracks (Mohd Faiz *et al.*, 2014; Purna *et al.*, 2012). Thermal oxidation process was chosen as surface treatment techniques on Co-Cr-Mo alloy due to no reports were found in elsewhere to explain the effects of HA coating with incorporation of oxide interlayer when tested in metal ions release test and cells behaviour. Meanwhile, this study also seeks to address the following questions:

i. How thermal oxidation temperature influences the formation of oxide interlayer properties?
ii. Can oxide interlayer help to reduce metal ions release when immersed in simulated body fluid?
iii. What the maximum adhesion strength of HA can be achieved on the oxidized Co-Cr-Mo alloy?
iv. How well is the cell grow on the HA coated with oxide interlayer samples as compared to without oxide interlayer samples?
v. What is the feasible slurry concentration for obtaining a good HA coating on Co-Cr-Mo alloy?
vi. What is the best sintering temperature on HA coated samples in order to avoid massive cracks?

1.3 Problem Statement

Co-Cr-Mo alloy implant is known to corrode over the time and start releasing harmful metal ions (Co, Cr, Mo) into body fluids (serum, urine and blood) once implanted and exposed to the aggressive body environment. The level of metal ions release and accumulation of wear particles can cause adverse clinical responses which affecting the stability of the implant (Roberto and Anna, 2011; Sun *et al.*, 2011). In order to overcome these problems, hydroxyapatite (HA) coatings can be introduced onto the metal surface to act as a barrier in reducing the release of excessive metal ions and also helps for bone to growth rapidly on the surface implants (Krishnamurithy, 2013; Liu *et al.*, 2004a; Ramaswamy *et al.*, 2009). It has also been reported that the survival rate of HA coated implants is high as compared to implants without HA coating (Kim *et al.*, 2004; Surmenev *et al.*, 2014).

There are various methods to coat HA on metal implants and one of them is sol-gel dip coating technique. This method have attracts most of researchers' attentions due to its ability to coat sample at room temperature and the thickness of coating can be controlled much easily as compared to plasma spray (Ben Naceur *et al.*, 2012; Fathi and Hanifi, 2007; Kim *et al.*, 2004; Zhang *et al.*, 2011). Despite many advantages of the sol-gel dip coating technique, there are several limitations exist arise. It has been reported that HA coating on metal implants often results in severe cracks and delamination which eventually lead to coating failure (Kirk and Pilliar, 1999; Roest *et al.*, 2011; Yang and Chou, 2007; Zhen-lin and Rong-chang, 2010). This phenomenon occurs due to poor adhesion strength between HA and the underlying substrate (Case *et al.*, 2005; Yang and Chou, 2007; Zhen-lin and Rong-chang, 2010) and the low cohesive strength of the coated material itself (Roest *et al.*, 2011; Zhen-lin and Rong-chang, 2010). It is also noted that the poor mechanical properties of HA such as brittleness and toughness have restrict its used in load bearing applications (Diangang *et al.*, 2008).

One of the solutions to overcome these issues is by introducing an intermediate layer in between the brittle HA coating and the metal substrate. It has been reported that the intermediate layer able to enhance the adhesive metal-ceramic (HA) bonding and coating integrity (García *et al.*, 2004; Kirk and Pilliar, 1999; Purna *et al.*, 2012; Rajesh *et al.*, 2011; Yang and Chou, 2007). Although the results showed promising in improving adhesion strength on other metallic biomaterials (Cao and Liu, 2013; Man *et al.*, 2009; Mohd Faiz *et al.*, 2014; Shaylin and George, 2012), research on Co-Cr-Mo alloy is somehow still limited especially involving the application of intermediate layer prior to HA coating. There is also lack of research studies in evaluating the performances of the oxide interlayer on Co-Cr-Mo alloy in reducing the release of metal ions and their responses to the cell growth.

In summary, much attention has been paid on HA coating as a solution to reduce metal ions release but not much research on the application of HA coating with intermediate layer on Co-Cr-Mo alloy. There is possibility that oxide interlayer (intermediate layer) created on Co-Cr-Mo alloy able to helps in reducing metal ions release. However, the performances of oxide interlayer in promoting better cell growth are still limited. Therefore, this research works is required in order to assess the effectiveness of oxide interlayer in the body fluid and so the evaluation has been justified.

1.4 Objectives of the Research

The principal objective of this research is to establish the methodology to improve HA coating on Cobalt-Chromium-Molybdenum (Co-Cr-Mo) alloy for biomedical implant application. The objectives of the research are as follows:

i. To evaluate the effects of thermal oxidation temperature on Co-Cr-Mo alloy surface morphology, adhesion strength and metal ions release.

ii. To evaluate the effects of HA slurry concentrations and sintering temperature on Co-Cr-Mo alloy surface morphology, adhesion strength and metal ions release.

iii. To evaluate the cell growth and cell attachment performances on HA coated with oxide interlayer on Co-Cr-Mo alloy.

1.5 Scopes of the Research

The research was conducted in the following limits:

i. Cobalt-Chromium-Molybdenum (Co-Cr-Mo) alloy was used as the substrate material.

ii. Thermal oxidation process was used to create oxide interlayer on Co-Cr-Mo alloy within temperatures range of 850°C to 1250°C at fixed time duration in atmospheric condition.

iii. HA coating was deposited on the samples using HTWL-01 Desktop Dip Coater (MTI Cooperation, USA) at room temperature.

iv. The adhesion strength of oxide interlayer and HA coating were measured using Revetest Scratch test in order to determine the critical load.

v. RPMI 1640 medium solution was used as the simulated body fluid for metal ions release tests up to 28 days.

vi. In-vitro biocompatibility of the HA coated samples was tested using Multipotent Stromal Cells (MSCs) up to 14 days for cell attachment study.

1.6 Significance of the Research

In this study, a development of a simple and effective surface treatment technique is applied on Co-Cr-Mo alloy to improve the bonding and quality of HA coating using sol-gel dip coating method. Preliminary results from other biomedical materials showed that HA coating via sol-gel dip coating method have been successfully improved biocompatibility, increase corrosion resistance and reduce metal ions release when tested in in-vitro tests. By achieving this purpose, hopefully future implants are much cheaper and affordable for anyone who are in needs. It is also hope that the outcomes of this research will help the country to reduce power consumption and lessen the expensive costs in producing medical implants. The development of systematic understanding on exploring this material might also help engineers and scientists to come up with better and efficient implants for future used.

1.7 Hypothesis of the Results

It is expected that good adhesion strength of oxide interlayer formed on Co-Cr-Mo alloy through thermal oxidation process is able to act as a barrier to reduce excessive metal ions release into simulated body fluid. It is also expected that HA coated samples with oxide interlayer exhibits better performances in cell attachment and cell proliferation as compared to HA coated samples without oxide interlayer. Theoretically, it is believed that with the combination of oxide interlayer and HA coating on Co-Cr-Mo alloy, it will be able to accelerate the spreading of cell growth thus, resulting in shortening the healing time after implantation.

1.8 Organization of Thesis

This thesis consists of 5 main chapters. Chapter 1 gives an overview of the research background and problem statements in this study. Chapter 2 provides the literature review on the current biomaterial implants used and work done to solve problems with Cobalt-Chromium-Molybdenum (Co-Cr-Mo) alloy. Information regarding existing techniques to coat HA on metal implants and current method used to examine the coating performances are also covered in this chapter. Methodology for the whole experiments is included in Chapter 3 starting from sample preparation, method used to run the experiments until the existing equipment involved in executing this research work. Chapter 4 consists of results gathered from the experimental work and discussion on the findings obtained. Lastly, Chapter 5 provides conclusions of the research work and some suggestions for future work.

CHAPTER 1

INTRODUCTION

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CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter discusses the approaches followed in conducting this research. The details of procedure, experimental plan and conditions for the experiments run as well as equipment's used including characterization methods are described in this chapter. This includes sample preparations, experimental set-up, mechanical testing methods and biocompatibility testing procedure.

3.2 Research Approach

In this research, the ultimate aim is to develop a cheaper yet effective method to coat hydroxyapatite (HA) using sol-gel dip coating technique and to improve the

HA bonding strength on the Co-Cr-Mo alloy by creating oxide interlayer at the sample surface. At the end of this research, bioactivity performances of HA coated samples with oxide interlayer were evaluated through in-vitro test such as metal ions release test and cell attachment responses. These tests were conducted to simulate its real applications as implant materials. Figure 3.1 summarizes the overall experiment flow whilst Figure 3.2 shows the details of experimental works.

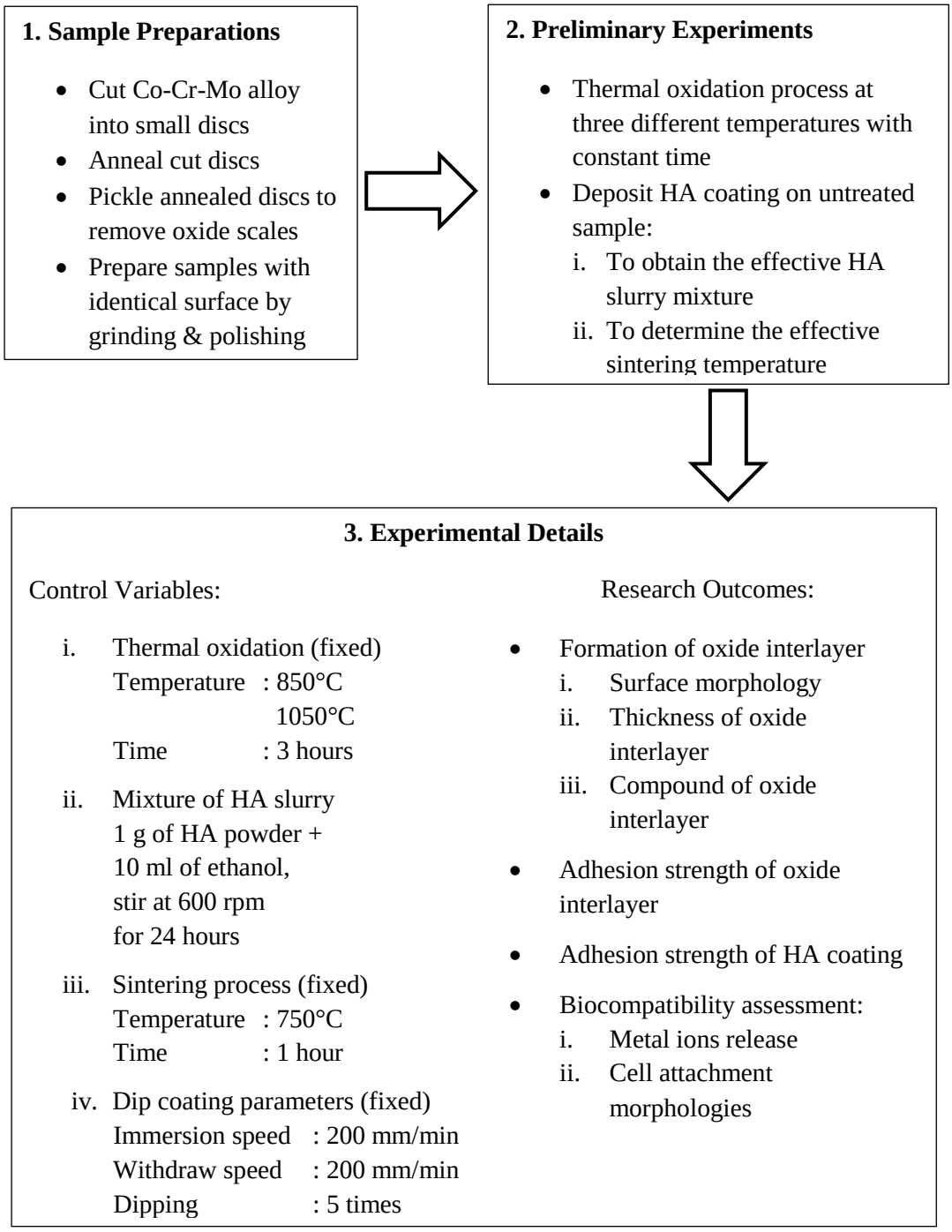

Figure 3.1 Box diagram of the overall experimental research methodology.

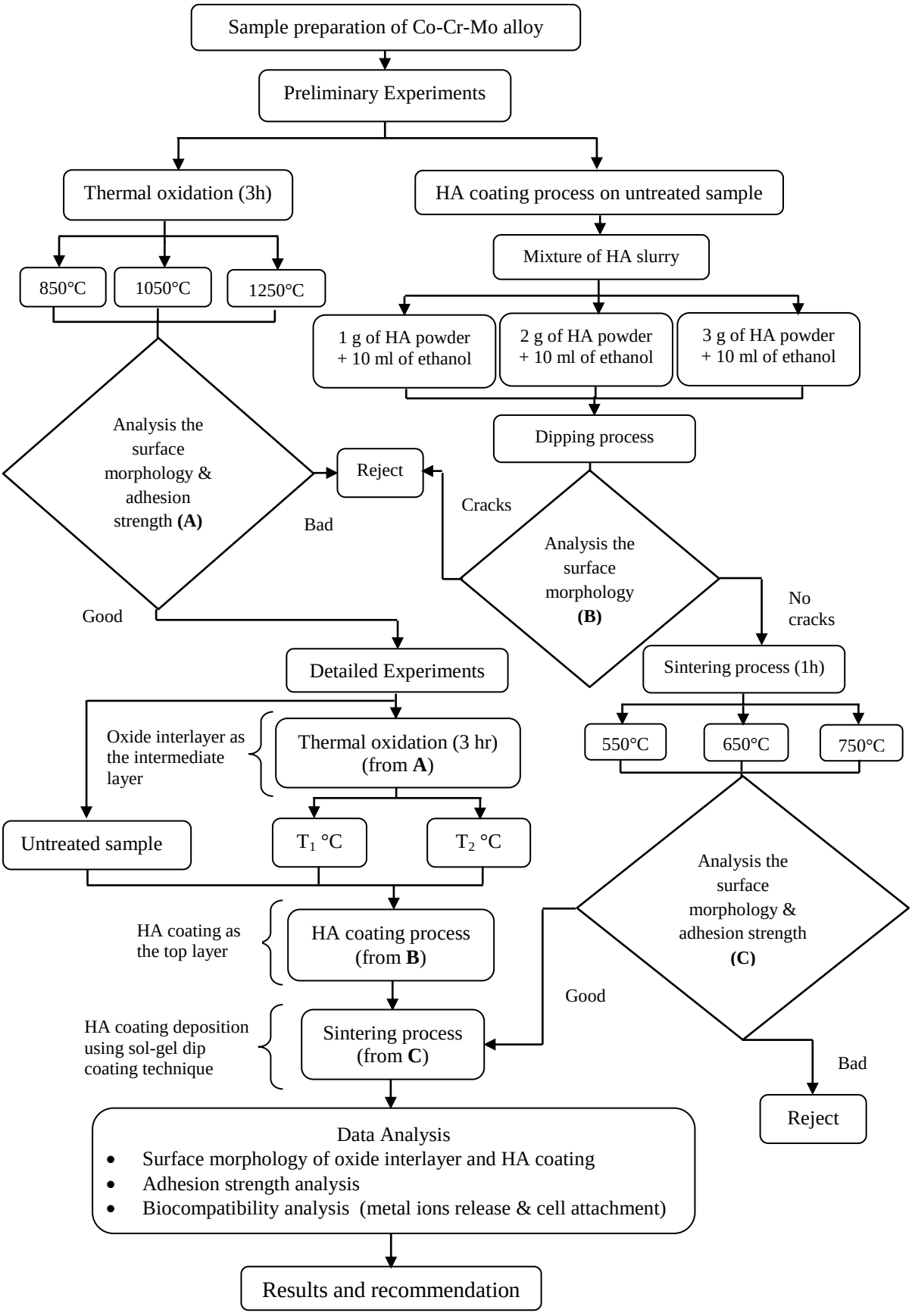

Figure 3.2 The overall flow chart of experimental works.