

**MICROSTRUCTURE AND PROPERTIES OF
SINTERED Co-Cr-Mo ALLOY POWDER UNDER
DIFFERENT PROCESSING CONDITIONS**

ZURAIĐAWANI BINTI CHE DAUD

UNIVERSITI MALAYSIA PERLIS

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**Microstructure and Properties of Sintered Co-Cr-Mo
Alloy Powder under Different Processing Conditions**

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2011

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Doctor of Philosophy

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UNIVERSITI MALAYSIA PERLIS**

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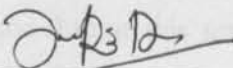
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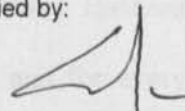
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LIST OF ABBREVIATIONS

Al	Aluminium
Al ₂ O ₃	Alumina
Ag	Silver
ASTM	American Standard Testing Method
Au	Gold
B ₄ C	Boron carbide
C	Carbon
CF	Carbon fiber
Ca ₁₀ (PO ₄) ₆ (OH) ₂	Hydroxylapatite
Ca ₂ P ₂ O ₇	Calcium pyrophosphate
Co	Cobalt
Co-Cr	Cobalt-Chromium
Co-Ni	Cobalt-Nickel
Co-Cr-Mo	Cobalt-Chromium-Molybdenum
Co-Ni-Cr-Mo	Cobalt-Nickel-Chromium-Molybdenum
Cr	Chromium
Cr ₂ O ₃	Chromium oxide
CuSO ₄	Copper (II) sulphate
Fe	Ferum (Iron)
H ⁺	Hydrogen ion
H ₂ O	Water

HCl	Hydrochloride
min	Minutes
Mn	Manganese
Mo	Molybdenum
Ni	Nickel
N _o	Rotation speed
O ₂	Oxygen
PEEK	polyethyletherketone
PM	Powder Metallurgy
PMMA	polymethymethacrylate
Pt	Platinum
PTFE	Polytetrafluoroethylene
PU	Polyurethane
RPM	Rotation Per Minute
SIT	Strain induced phase transformation
Si	Silicate
Si ₃ N ₄	Silicon nitride
SiC	Silicon carbide
Ti	Titanium
Ti-6Al-4V	Titanium-6Aluminum-4Vanadium
UHMWPE	Ultra High Molecular Weight Polyethylene
V	Vanadium
W	Tungsten

ZrO ₂	Zirconia
fcc	Face centred cubic
hcp	Hexagonal close packed
MS	Metalurgi Serbuk
PPM	Putaran Per Minit
75% H ₂ -25% N ₂	75% Hydrogen-25% Nitrogen

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LIST OF SYMBOLS

°C	Degree Celsius
%	Percent
wt. %	Weight percent
cm ²	Centimetre square
HV	Hardness Vickers
g	Gram
g/cm ³	Gram per centimetre cube
GN/m ²	Gega Newton per metre square
J/mol-K	Joule per mol-Kelvin
KN	Kilo Newton
kJ/mol	Kilo joule per mol
mg/cm ²	Milligram per centimetre square
min	Minutes
mm/min	Millimetre per minutes
mpy	Mil per year
MPa	Mega Pascal
Q	Activation energy
µm	Micrometer

Microstructure and Properties of Sintered Co-Cr-Mo Alloy Powder under Different Processing Conditions

ABSTRACT

Co-Cr-Mo (ASTM F-75) alloy is one of the most important metallic biomaterials that are commonly used for surgical implant due to its mechanical properties, good wear resistance and biocompatibility. This study has focused on the effect of sintering time and sintering temperature on the microstructure development and properties of sintered Co-Cr-Mo alloy powder, sintering mechanism and activation energy, and corrosion behaviour. In the fabrication of PM Co-Cr-Mo alloy, five weight percentages (wt. %), 1.0, 1.5, 2.0, 2.5 and 3.0 of binder (stearic acid) were studied to determine the optimum amount of binder content based on the results of linear shrinkage, bulk density, apparent porosity and Vickers microhardness of the sintered samples. Then the Co-Cr-Mo alloy powder were blended with the selected amount of optimum binder (2wt. % of stearic acid) using a rotation mill at 165RPM for 30 minutes, uniaxially pressing at 500MPa and sintering in a furnace at three different sintering temperatures (1250°C, 1300°C and 1350°C) for five sintering times (30, 60, 90, 120 and 150 minutes) in argon atmosphere. The characterisation on sintered samples were carried out based on microstructure, grain size, bulk density and apparent porosity, Vickers microhardness test and followed by compressive strength. The study of sintering mechanism was carried out in order to determine the activation energy of Co-Cr-Mo alloy. The corrosion behaviour of selected samples was analysed based on the minimum value of compressive strength. For corrosion test, the selected samples were immersed in simulated body fluid, 0.9% sodium chloride (NaCl) solution at 37°C for 90 days. From this study, the values of bulk density and grain size increased with increasing sintering temperature and sintering time. The bulk density values are in the range 7.04g/cm³ to 7.21 g/cm³, 7.16 g/cm³ to 7.28 g/cm³ and 7.45 g/cm³ to 7.54 g/cm³ for sintering temperature of 1250°C, 1300°C and 1350°C, respectively for five sintering times. Meanwhile, the grain sizes for five sintering times are 25.6µm to 37.7µm, 36.6µm to 44.5µm and 80.4µm to 89.9µm respectively for the three sintering temperatures. However, opposite results were obtained for apparent porosity, hardness and compressive strength. The samples sintered at 1350°C have the highest values of hardness (303HV-294HV) and compressive strength (329MPa-206MPa) for 30 to 150 minutes of sintering times. Based on the fracture mode, all samples show the fracture with a shear mode and occurred close to an angle of 45° from the compressive axis. The samples sintered at 1250°C and 1300°C exhibited smooth transgranular fracture mode. Meanwhile, the step-like transgranular fracture mode was observed in the samples sintered at 1350°C. The results of corrosion test showed that sample sintered at 1300°C gives the highest value of corrosion rate (0.075mpy) meanwhile sample sintered at 1350°C has the lowest corrosion rate (0.006mpy). From this study, the samples sintered at 1350°C with 120 minutes of sintering times showed the compressive strength close to the bone strength and better corrosion properties.

Mikrostruktur dan Sifat-Sifat bagi Serbuk Aloi Co-Cr-Mo Tersinter dibawah Keadaan Pemrosesan yang Berbeza

ABSTRAK

Co-Cr-Mo (ASTM F-75) aloi merupakan bahan bio logam yang sangat penting yang biasanya digunakan untuk implan disebabkan oleh sifat mekanik, ketahanan haus yang sangat baik dan keserasian bio. Kajian ini dijalankan dengan tumpuan kepada kesan suhu dan masa persinteran terhadap pertumbuhan mikrostruktur dan sifat-sifat serbuk aloi Co-Cr-Mo tersinter, mekanisme persinteran dan tenaga pengaktifan, dan kelakuan kakisan. Dalam fabrikasi MS Co-Cr-Mo aloi, lima peratusan berat (1.0, 1.5, 2.0, 2.5 dan 3.0) bahan pengikat (asid stearik) dikaji untuk menentukan kandungan bahan pengikat yang optimum berdasarkan beberapa ujian seperti ujian pengecutan linear, ketumpatan pukal, peratus keliangan dan juga ujian kekerasan mikro. Kemudian, serbuk aloi Co-Cr-Mo diadun dengan 2 peratus berat asid stearik (peratusan optimum) dengan menggunakan mesin putaran pada 165PPM selama 30 minit, penekanan searah pada 500MPa dan disinter di dalam relau pada tiga suhu yang berbeza (1250°C, 1300°C dan 1350°C) pada lima masa persinteran yang berbeza (30, 60, 90, 120 dan 150 minit) dalam atmosfera argon. Pencirian pada setiap sampel dijalankan berdasarkan mikrostruktur, saiz butiran, ketumpatan pukal, keliangan ketara, kekerasan dan kekuatan mampatan. Kajian mekanisme persinteran juga dijalankan untuk menentukan tenaga pengaktifan Co-Cr-Mo aloi. Kelakuan kakisan bagi sampel yang terpilih dianalisis berdasarkan nilai minimum kekuatan mampatan. Bagi ujian kakisan, sampel yang tertentu direndam di dalam bendalir badan tersimulasi, 0.9% natrium klorida (NaCl) pada suhu 37°C selama 90 hari. Daripada kajian ini, nilai ketumpatan pukal dan saiz butir meningkat dengan peningkatan suhu dan masa persinteran. Nilai ketumpatan pukal adalah dalam julat 7.04 g/cm³ sehingga 7.21 g/cm³, 7.16 g/cm³ sehingga 7.28 g/cm³ dan 7.45 g/cm³ sehingga 7.54 g/cm³ untuk suhu persinteran 1250°C, 1300°C dan 1350°C masing-masing dengan lima masa persinteran. Manakala saiz butiran untuk lima jenis masa persinteran adalah dalam julat 25.6µm hingga 37.7µm, 36.6µm hingga 44.5µm dan 80.4µm hingga 89.9µm masing-masing untuk tiga suhu persinteran yang berlainan. Namun begitu, keputusan sebaliknya terhasil untuk ujian keliangan ketara, kekerasan dan kekuatan mampatan. Sampel yang disinter pada suhu 1350°C memiliki nilai kekerasan yang paling tinggi (303-294) dan kekuatan mampatan (329MPa-206MPa) bagi 30 hingga 150 minit masa persinteran. Berdasarkan mod patah, semua sampel mempamerkan mod ricih yang terjadi pada sudut 45° dari paksi mampatan. Sampel yang disinter pada suhu 1250°C dan 1300°C mempamerkan mod patah licin transgranular. Sementara sampel 1350°C menunjukkan mod patah transgranular bertingkat. Keputusan kakisan menunjukkan bahawa sampel yang disinter pada suhu 1300°C mempunyai kadar kakisan yang tinggi (0.075mpy) manakala sampel yang disinter pada suhu 1350°C mempunyai kadar kakisan yang paling rendah (0.006mpy). Daripada kajian ini, sampel yang disinter pada suhu 1350°C dengan masa persinteran 120 minit menunjukkan kekuatan mampatan yang hampir dengan kekuatan tulang dan sifat kakisan yang paling baik.

CHAPTER 1

INTRODUCTION

1.1 Introduction

For more than a generation, various materials so-called biomaterials are used in medicine and dentistry with a purpose to replace or repair a body feature, tissue, organ or function. The performance of biomaterial in direct contact with living tissue is controlled by two sets of characteristics: biofunctionality and biocompatibility (Matković et al., 2004). Biomaterials is defined as materials of natural or manmade origin that used to direct, supplement, or replace the functions of living tissues of human body (Park & Lakes, 1992). Ramakrishna et al. (2001) have reported that the uses of biomaterials as artificial eyes, ears, teeth and noses were found since Egyptian mummies. The advancement of many fields of technology is conditioned by acquisition of materials with ever increasing performance. Therefore an effort to improve the properties of new and existing materials has been receiving attention across the globe for number of year. Current medical practice used a large number of devices and implants.

Biomaterials in term of implants (dental implants, orthopaedic implants, sutures, bone plates, joint replacement, ligaments, vascular graft, heart valve, etc.) and medical devices (pacemakers, biosensors, artificial heart, blood tubes, etc.) are widely used to replace or restore the function of traumatized or degenerated tissues or organ, to assists in healing, to improve function, to correct abnormalities, and thus improve the