Development and Characterization of Co-Cr-Mo (F-75 alloy)/hydroxyapatite Composites Fabricated by Powder Metallurgy for Biomedical Applications

By

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LIST OF ABBREVIATIONS

A/W	Apatite/Wollastonite
AISI 316L	0.03 wt. % C, 17-20 wt. % Cr, 12-14 wt. % Ni, 2-3 wt. % Mo and minor amounts of nitrogen, manganese, phosphorus, silicon and sulphur
ASTM	American Society for Testing and Materials
AZ91-HAP	Magnesium-Hydroxyapatite
B_4C	Boron carbide
C	Carbon
$Ca_2P_20_7$	Calcium Pyrophosphate
$CaCO_3$	Calcium Carbonate
$CaHPO_4.2H_2O$	Calcium Hydrogen Phosphate
$Ca_{10}(PO_4)_6(OH)_2$	Hydroxyapatite
CeO_2	Ceria
CMC	Ceramic matrix composites
Co	Cobalt
CO_2	Carbon dioxide
Co-Cr	Cobalt-Chromium
Co-Cr-Mo	Cobalt-Chromium-Molybdenum
Cr	Chromium
CR	Corrosion Rate
E_{corr}	Corrosion current
EW	Equivalent weight

FA	Fluoroapatite
FCC	Face centre cubic
Fe	Ferum
H_2O	Water
HAP	Hydroxyapatite
HCAp	Hydroxyl carbonate apatite
HCP	Hexagonal close-packed
Mg	Magnesium
MMC	Metal Matrix Composites
Mn	Manganese
Mo	Molybdenum
Ni	Nickel
Ni Ti	Nickel-titanium
OH^-	Hydrogen ion
PE	Polyethylene
PGA	Polyglycolide
PLA	Polylactic acid
PM	Powder Metallurgy
PMC	Polymer matrix composite
PMMA	Polymethyl-methacrylate
PP	Polypropylene
Pt	Platinum
R_p	Polarization resistance ($\Omega.cm^2$).
rpm	Rotation Per Minute
SBF	Simulated Body Fluid

Si	Silicon
SiC	Silicon carbide
Si_3N_4	Silicon Nitride
SMA	Shape Memory Alloy
TAP	Transferred Arc Plasma
Ti	Titanium
Ti-6Al-4V-HAP	Titanium-6Aluminum-4Vanadium-Hydroxyapatite
Ti-HAP	Titanium-Hydroxyapatite

LIST OF SYMBOLS

%	Percent
$\Omega.cm^2$	Ohm centimetre square
^0C	Degree Celsius
A	exposed specimen area, cm^2
g/cm^3	gram per centimetre cube
GN/m^2	Giga Newton per metre square
GPa	Giga Pascal
KN	Kilo Newton
min	minutes
MPa	Mega Pascal
mpy	Mil per year
N_0	Rotational speed
wt. %	Weight Percent
µm	micrometer

Pembangunan dan Pencirian Komposit Co-Cr-Mo (Aloi F-75)/Hidroksiapatit yang Difabrikasi Menggunakan Metalurgi Serbuk Untuk Aplikasi Bioperubatan

ABSTRAK

Aloi Co-Cr-Mo (F-75) telah terkenal digunakan dalam bidang bioperubatan kerana biokeserasian yang amat baik apabila digunakan di dalam badan manusia atau haiwan. Serbuk hidroksiapatit (HAP) telah digunakan sebagai bahan pengisi kerana HAP adalah salah satu bahan yang mempunyai biokeserasian yang amat efektif bersamaan dengan kandungan mineral untuk tulang dan gigi. Kajian ini melaporkan tentang fabrikasi dan pencirian aloi F-75 ditambah dengan HAP yang disediakan menggunakan kaedah metalurgi serbuk. Kajian ini dijalankan dengan tumpuan kepada kesan penambahan HAP kepada aloi F-75 dan suhu persinteran terhadap sifat-sifat fizikal dan mekanikal komposit F-75/HAP, mikrostruktur, serta kelakuan kakisan dan bio-aktiviti komposit ini. Dalam fabrikasi komposit F-75/HAP, 2, 4, 6, 8 dan 10 % berat HAP telah ditambah kepada aloi F-75. Sampel rujukan aloi F-75 (tanpa penambahan HAP) juga telah disediakan untuk semua suhu persinteran. Campuran diadun menggunakan mesin putaran selama 20 minit pada 154 rpm sebelum ditekan sejuk pada 550 MPa menggunakan mesin penekanan searah. Sampel disinter pada tiga suhu persinteran yang berbeza (1100^{0}C, 1150^{0}C dan 1200^{0}C) di dalam relau tiub selama 2 jam. Sifat-sifat fizikal ditentukan melalui ujian ketumpatan pukal dan keliangan ketara, manakala sifat mekanikal ditentukan melalui ujian kekuatan mampatan. Kelakuan kakisan komposit F-75/HAP dianalisis menggunakan ujian elektrokimia yang dikawal oleh potensiostat Gamry G300. Kelakuan bio-aktiviti komposit ini telah dijalankan secara in-vitro dengan merendam komposit ke dalam bendalir badan tersimulasi selama 18 hari. Analisis XRD, SEM, FTIR dan pH dilakukan untuk menentukan kehadiran lapisan apatit di permukaan komposit F-75/HAP. Daripada kajian ini, nilai ketumpatan pukal berkurang apabila kandungan HAP bertambah. Nilai tertinggi ketumpatan pukal adalah pada komposit yang mengandungi 2% berat HAP dengan ketumpatan 6.6217 g/cm^3 dengan suhu persinteran 1200^{0}C, manakala nilai ketumpatan pukal terendah adalah pada komposit dengan penambahan 10% berat HAP selepas disinter pada suhu 1150^{0}C (4.3915 g/cm^3). Keliangan ketara sampel yang disinter menunjukkan bahawa apabila penambahan HAP ditingkatkan, keliangan ketara akan meningkat di dalam julat 13.13% (untuk 2% berat HAP) ke 37.58% (untuk 10 % berat HAP). Kekuatan mampatan berkurang dengan penambahan HAP. Sampel dengan penambahan 2 % berat HAP dengan suhu persinteran 1200^{0}C, memberi nilai kekuatan mampatan yang paling tinggi (341.81 MPa). Mikrostruktur komposit F-75/HAP selepas disinter pada tiga suhu persinteran yang berbeza menunjukkan keliangan dan kegumpalan HAP meningkat mengikut suhu persinteran. Keputusan ujian kakisan menunjukkan sampel dengan penambahan 8 % berat HAP memberikan nilai kadar kakisan yang paling rendah (16.59 x 10^{-6} mpy untuk F-75/8%HAP yang disinter pada 1150^{0}C). Daripada keputusan ujian bio-aktiviti, lapisan apatit karbonat telah terbentuk pada permukaan komposit. Berdasarkan keputusan ujian sifat fizikal dan mekanikal komposit, penambahan HAP yang optimum terhadap aloi F-75 adalah 2 % berat, manakala sampel yang disinter pada suhu tinggi (1200^{0}C) menunjukkan sifat fizikal, mekanikal dan juga kelakuan kakisan yang baik. Daripada ujian kakisan, komposit F-75/6%HAP dan F-75/8%HAP yang disinter pada suhu tinggi menghasilkan kerintangan kakisan yang baik. Aloi F-75 yang biolengai boleh ditukarkan kepada bioaktif dengan menambahkan sehingga 10 % berat HAP.

Development and Characterization of Co-Cr-Mo (F-75 alloy)/hydroxyapatite Composites Fabricated by Powder Metallurgy for Biomedical Applications

ABSTRACT

Co-Cr-Mo (F-75) alloy is known to be used in biomedical field because of their excellent biocompatibility when implanted to human or animal body. Hydroxyapatite (HAP) powders have been used as filler because HAP is the one of the most effective biocompatible materials with similarities to mineral constituents of bones and teeth. This research reported the fabrication and characterization of F-75 alloy filled with HAP which have been prepared by powder metallurgy method. This study has focused on the effect of HAP addition into F-75 alloy and sintering temperature on the physical and mechanical properties of the F-75/HAP composites, its microstructure, and also its corrosion and bioactivity behaviour. In fabrication of the F-75/HAP composite, 2, 4, 6, 8 and 10 wt. % of HAP have been added to F-75 alloys. The reference samples of F-75 alloy (with no addition of HAP) also have been prepared for all sintering temperatures. The mixtures were milled on a rotation mill for 20 minutes at 154 rpm before cold compacted at 550 MPa using an uniaxial press machine. The samples then have been sintered at three different sintering temperatures (1100^0C, 1150^0C and 1200^0C) in a tube furnace for 2 hours. Physical properties were measured by means of bulk density and apparent porosity while mechanical property was measured in term of compressive strength. The corrosion behaviour of the F-75/HAP composite has been analysed using electrochemical test controlled by Gamry G300 potentiostat. Bioactivity test for the composite was conducted in-vitro by immersing the composite into simulated body fluid for 18 days. XRD, SEM, FTIR and pH analyses had been done in order to observe the presence of the apatite layer on the surface of F-75/HAP composites. From this study, the values of bulk density decreased as the HAP content increased. The highest value of bulk density was gained by the composite with 2 wt. % of HAP with value 6.6217 g/cm^3 with sintering temperature 1200^0C, while the lowest bulk density value was given by the composite with 10 wt. % of HAP after sintered at 1150^0C (4.3915 g/cm^3). The apparent porosity was increased in the range of 13.13% (for 2 wt. % HAP) to 37.58% (for 10 wt. % HAP). Compressive strength was decreased by the additional of HAP. The sample with 2 wt. % of HAP addition with sintering temperature 1200^0C gave the highest compressive strength (341.81 MPa). The microstructure of F-75/HAP composites after sintering at three different sintering temperatures showed that porosity and HAP agglomeration increased with HAP content and sintering temperature. The results of corrosion test showed that the samples with 8 wt. % HAP addition gave the lowest value for corrosion rate (16.59 x 10^{-6} mpy for F-75/8% HAP sintered at 1150^0C). From bioactivity test results, the carbonated apatite layer was formed on the surfaces of the composite. According to the results for physical and mechanical properties testing of the composites, the optimum HAP addition to F-75 alloy was 2 wt. %, while samples that have been sintered at higher temperature (1200^0C), showed good physical and mechanical properties and also corrosion behavior. From corrosion test, F-75/6% HAP and F-75/8% HAP composites that have been sintered at higher temperature showed good corrosion resistance. Bioinert F-75 alloys can be converted into F-75 bioactive type by adding up to 10 wt. % of HAP.