THERMAL OXIDATION AND HYDROXYAPATITE COATING OF SLMed 316L STAINLESS STEEL SUBSTRATES

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Master of Science

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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LIST OF SYMBOLS

%	Percentage
°	Degree
2Ө	2 thetha
Å	Angstrom
CR	Corrosion rate (mm/yr)
D_{10}	Range of particle size diameter at 10 %
D_{50}	Range of particle size diameter at 50 %
D_{90}	Range of particle size diameter at 90 %
Ecorr	Potential current density (mV vs SCE)
EW	Equivalent weight
Icorr	Corrosion current density (mA/cm^2)
K	Constant (3.27×10^{-3} mmg/µAcmyr)
λ	Wavelength

LIST OF ABBREVIATIONS

Al_2O_3	Aluminium dioxide
Δ-Fe	Delta ferrite
316L SS	316L stainless steel
3D	Three dimensional
AM	Additive manufacturing
ASTM	American Society for Testing and Materials
CAD	Computer aided design
Ca-P	Calcium phosphate
Cr_2O_3	Chromium oxide
EDM	Electrical discharge machining
EDX	Energy dispersive X-ray
FDA	Food and Drug Administration
Fe_2O_3	Iron oxide
FESEM	Field emission scanning electron microscope
HAp	Hydroxyapatite
ICDD	The International for Diffraction Data
ISO	The International Organization for Standardization
OCP	Open-circuit potential
OM	Optical microscope
PMMA	Poly(methyl methacrylate)
SCE	Saturated calomel electrode
SEM	Scanning electron microscopy
SLM	Selective laser melting
TCP	Tricalcium phosphate
THA	Total Hip Arthroplasty
TiO_2	Titanium oxide
TTCP	Tetracalcium phosphate
UK	United Kingdom
USA	United State of America
XRD	X-ray Diffraction
ZrO_2	Zirconium oxide
γ-Fe	Austenite

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ABSTRAK

Keluli tahan karat 316L (316L SS) merupakan satu daripada biomaterial logam yang telah digunakan sebagai implan logam di dalam bidang bioperubatan sebagai pengganti atau pemulihan fungsi tisu-tisu atau organ-organ yang merosot. Dalam kajian ini, 316L SS telah dipilih sebagai substrat logam dan difabrikasikan dengan menggunakan teknologi-teknologi pembuatan budaya tambah (AM) melalui kaedah teknik laser lebur (SLM). Bersandarkan masalah kakisan 316L SS, pengoksidaan haba (TO) dan penyalutan *hydroxyapatite* (HAp) telah dilakukan dalam kajian ini. TO telah dijalankan pada 700 °C selama 150 jam, 200 jam dan 250 jam untuk membentuk lapisan oksida. 316L SS *SLMed* yang terma teroksida telah diselidiki berdasarkan pencirian permukaan dan sifat kakisan. Keadaan optimum bagi TO diteruskan dengan penyalutan HAp. Substrat 316L SS *SLMed* yang tidak teroksida dan terma teroksida disaluti *sol-gel* penyalutan celup dan disinter selama 1 jam pada 500 °C. Kesan daripada TO dan penyalutan HAp telah dikaji ke atas substrat 316L SS. Peratus berat substrat dan ketebalan lapisan oksida telah meningkat dengan peningkatan masa dedahan semasa TO. Kemunculan besi oksida (Fe_2O_3) sebagai lapisan oksida telah dikesan pada substrat terma teroksida. Namun begitu, kromium oksida (Cr_2O_3) telah dikesan pada substrat terma teroksida selama 150 jam. Dalam kajian ini, hanya TO 150 jam menunjukkan penambahbaikan dalam sifat kakisan kerana kehadiran lapisan-lapisan Fe_2O_3 dan Cr_2O_3. Masa pendedahan yang lama menyebabkan tiada perubahan dalam sifat kakisan. Oleh yang demikian, terma teroksida 316L SS SLMed telah diteruskan kepada penyalutan HAp. Salutan HAp ke atas terma teroksida 316L SS SLMed menujukkan salutan tebal dan bebas retakan. Berdasarkan keputusan kakisan, ianya terbukti bahawa kehadiran lapisan oksida dan salutan HAp mampu meningkatkan sifat kakisan terhadap 316L SLMed. Kadar kakisan bagi substrat 316L SS yang bersalut ternyata paling rendah berbanding kedaan substrat yang lain. Ternyata, kehadiran lapisan-lapisan oksida bagi substrat bersalut terma teroksida menunjukkan perbezaan ketara dalam sifat kakisan kerana kehadiran Cr_2O_3 dan Fe_2O_3 sebagai lapisan oksida. Kesimpulannya, lapisan-lapisan oksida dan salutan HAp yang terbentuk pada substrat 316L SS terma teroksida membantu dalam meningkatkan sifat kakisan untuk substrat 316L SS SLMed. Dengan kemunculan kaedah fabrikasi yang baru untuk biomaterial logam bersama lapisan oksida dan penyalutan HAp ternyata mampu membaik pulih kadar kakisan 316L SS. Nilai-nilai kadar kakisan bagi semua keadaan 316L SLMed juga boleh diterima berbanding dengan kaedah fabrikasi konvensional yang lain.

ABSTRACT

316L Stainless Steel (316L SS) is one of the metallic biomaterials that commonly applied as a metal implant in the biomedical field as substitute or function restoration of degenerated tissues or organs. In this study, 316L SS taken as the metal substrate and fabricated through to additive manufacturing (AM) technology which is via selective laser melting (SLM) process. Concerning the corrosion issue of 316L SS, thermal oxidation (TO) and hydroxyapatite (HAp) coating were introduced in this study. The TO was conducted at 700 °C for 150, 200 and 250 h to develop oxide layer formation. The thermally oxidised SLMed 316L SS substrates were investigated based on a surface characterisation and a corrosion behaviour. The optimum condition for TO then continues with the HAp coating application. Sol-gel dip coating coated the non-oxidised and thermally oxidised SLMed 316L SS substrates and sintered at 500 °C for 1 h. The effect of TO and HAp coating were examined on the SLMed 316L SS substrates. The weight percentage of the SLMed substrates and the thickness of oxide layer increased with the increment of soaking time during TO. The iron oxide (Fe_2O_3) was detected as the oxide layer on thermally oxidised substrates. However, chromium oxide (Cr_2O_3) was detected on thermally oxidised for 150 h. In this study, only TO for 150 h showed improvement in corrosion behaviour due to the presence of Fe_2O_3 and Cr_2O_3 layers. Prolonged soaking time shows no improvement in corrosion behaviour. Consequently, the thermally oxidised SLMed 316L SS substrates for condition 150 h have proceeded to coat with HAp. The HAp coating on thermally oxidised SLMed 316L SS substrates exhibited thicker coating deposition and free crack coatings surface compared to coated non-oxidised SLMed substrates. Based on corrosion result, it confirmed the formation of the oxide layer and HAp coating able to enhance corrosion resistance of the SLMed 316L SS. The corrosion rate for coated thermally oxidised SLMed substrates is the lowest compared to other substrate conditions. The presence of oxide layers on the coated thermally oxidised SLMed 316L SS substrates show a significant difference in the corrosion behaviour due to the presence of Cr_2O_3 and Fe_2O_3 layers. It concluded that the oxide layers and HAp coating formed on thermally oxidised SLMed substrate gave full support to enhance corrosion behaviour of the SLMed substrate. Introducing new fabrication method for metallic biomaterials with presence of oxide layer and HAp coating was able to improve corrosion behaviour of 316L SS. The values of corrosion rate for all SLMed 316L conditions were also acceptable and tolerable compared to others conventional fabrication methods.

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