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A STUDY AND DEVELOPMENT OF COATING TITANIUM DIOXIDE FILMS ON  
316L STAINLESS STEEL BY A DUAL CATHODE DC UNBALANCED  
MAGNETRON SPUTTERING FOR MEDICAL APPLICATIONS

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A Dissertation Submitted in Partial Fulfillment of the Requirements for  
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หัวข้อวิทยานิพนธ์	การศึกษาและพัฒนากลือบฟิล์มไททาเนียมไดออกไซด์บนสแตนเลส สตีลเกรด 316L โดยวิธี ดูโอ แคโทด ดีซี อัลตราซันเมกนีตรอนสปัตเตอริง สำหรับประยุกต์ใช้ทางการแพทย์
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## บทคัดย่อ

การเกิดอุบัติเหตุเกี่ยวกับกระดูกส่งผลกระทบต่อคุณภาพชีวิตของผู้ประสบเหตุ ในหลายๆ ปีที่ผ่านมามีการใช้โลหะ และโลหะผสมสำหรับใช้แทนที่หรือรักษากระดูกที่เกิดอุบัติเหตุ ซึ่งโลหะที่ใช้จะมีอยู่ด้วยกัน 3 ชนิด คือ สแตนเลส สตีล, โคบอลต์-โครเมียม และ ไททาเนียม รวมถึงโลหะผสมของไททาเนียม ในจำนวนโลหะทั้ง 3 ชนิด สแตนเลส สตีล มีการใช้งานมากที่สุด เนื่องจากผลิตง่าย และมีราคาถูก เมื่อเทียบกับโลหะชนิดอื่นๆ แต่การใช้สแตนเลส สตีล เป็นเวลานานจะเกิดการปลดปล่อยไอออนอิสระออกมาสู่ร่างกาย และทำให้เกิดผลเสียต่อสุขภาพ

รูไทท์โลหะไททาเนียมเป็นโลหะที่มีความสามารถเข้าได้ดีกับเนื้อเยื่อของร่างกาย โดยที่ผิวของไททาเนียมจะถูกทำให้กลายเป็นไททาเนียมไดออกไซด์ เพื่อเพิ่มประสิทธิภาพการเนื้อมาให้เกิดเซลล์กระดูกบนผิวของไททาเนียม แต่ไททาเนียมเป็นโลหะที่มีราคาแพงมาก และไททาเนียมไดออกไซด์ก็ไม่สามารถเกิดขึ้นได้เองภายใต้สภาวะของร่างกายของสิ่งมีชีวิต ดังนั้นจึงต้องทำการเคลือบผิวด้วยไททาเนียมไดออกไซด์ก่อน

ฟิล์มไททาเนียมไดออกไซด์ถูกสังเคราะห์ด้วยดูโอแคโทด ดีซี อัลตราซันเมกนีตรอนสปัตเตอริงภายใต้สภาวะการเปลี่ยนแปลงเวลาเคลือบ, ระยะทางระหว่างเป้ากับผิวเคลือบ และการใช้ความต่างศักย์ไบแอสที่ผิวเคลือบ พบว่าความขรุขระ, ความหนา และขนาดเกรนจะเพิ่มขึ้นเมื่อเพิ่มเวลาในการเคลือบ และลดระยะห่างระหว่างเป้ากับผิวเคลือบลง มากไปกว่านั้นระยะนาบของ (110) รูไทท์ สามารถเปลี่ยนไปเป็น (101) รูไทท์ เมื่อทำการเพิ่มความต่างศักย์ไบแอสที่ผิวเคลือบ

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สำหรับการทดลองจำลองการปลูกกระดูก (สารประกอบอปาไทต์) พบว่าพื้นที่ผิว และโครงสร้างของผิวมีผลต่อการเกิดสารประกอบอปาไทต์ โดยสารประกอบอปาไทต์สามารถเกิดได้ง่ายบนพื้นผิวที่มีพื้นที่ผิวมาก และมีโครงสร้างที่อยู่ระหว่าง (110) รูไทต์ และ (101) รูไทต์

คำสำคัญ:  $\text{TiO}_2$ / รูไทต์/ อปาไทต์/ SBF/ ฟิล์มบาง

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### Abstract

Bone damage is a serious health condition that has a direct impact on the quality of life of sufferers. In recent years, metals and alloys are widely used for restoration of bone damage. However, the degradation of most metals implanted in human body has narrowed the choice of clinically usable metals and alloys to mainly stainless steel, cobalt-chromium and titanium and its alloys. Among all the metallic materials, the austenitic stainless steel type 316L are the most popular materials because of their relatively low cost, ease of fabrication and reasonable corrosion resistance. However, clinical experience has shown that they are susceptible the localized attack in long-term.

Titanium and its alloys are thought to be highly biocompatible materials. The excellent biocompatibility of titanium and its alloys is associated with the properties of their protective surface oxide, titanium dioxide ( $\text{TiO}_2$ ). However, the natural oxide films may not be protective enough in the aggressive biologic environment, and there have recently been some clinical papers reporting hypersensitivity and allergic reactions to titanium. One way to solve this problem is to coat  $\text{TiO}_2$  thin films directly and test *in vitro*.

Rutile  $\text{TiO}_2$  thin films were deposited using dual cathode dc unbalanced magnetron sputtering with various deposition times, target to substrate distance ( $d_{t-s}$ ) and applied substrate bias voltage ( $V_{sb}$ ) to investigate the structure of  $\text{TiO}_2$  films. It was found that the surface roughness, thickness and average grain size of rutile  $\text{TiO}_2$  films increased with decreasing the  $d_{t-s}$  and increasing the coating times. Moreover, the (110) rutile plane shift to (101) plane with increasing the  $V_{sb}$ .

*In vitro*, the apatite can be found on all samples. However, the surface area and the surface structure were affected on the formation of apatite layer. The apatite easy formed on the high surface area and balanced plane of (110) and (101) properties.

Keywords:  $\text{TiO}_2$ / Rutile/ Apatite/ SBF/ Thin Film

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LIST OF SYMBOLS

A	Apatite
Ar	Argon
Ar <sup>+</sup>	Argon ion
cm	Centimeter
°C	Degree Clausius
d <sub>t-s</sub>	Distant of target to substrate
R	Rutile
SBF	Simulated body fluid
Ti	Titanium
Ti <sup>+</sup>	Titanium ion
TiO <sub>2</sub>	Titanium dioxide
V <sub>sb</sub>	Substrate bias voltage

## LIST OF ABBREVIATIONS

2D	Two-dimensional
3D	Three-dimensional
AFM	Atomic force microscopy
mtorr	Milli-torr
SEM	Scanning electron microscopy
XRD	X-ray diffraction