AN ABSTRACT OF THE THESIS OF

Nicole Lea Bauer for the degree of Honors Baccalaureate of Science in Chemical Engineering presented on April 22nd, 2011. Title: Nanomechanical Characterization of Atomic Layer Deposition Coatings for Biomedical Applications

Abstract approved: _____

Dr. John Conley

Abstract approved: _____

Dr. John Mitchell

Atomic Layer Deposition (ALD) is a promising technique for the production of biologically safe, wear resistant and corrosion protective coatings for orthopedic applications. In this work, the impact of coating thickness and surface preparation on the hardness (H), elastic modulus (E), wear resistance, and delamination of ALD Al_2O_3 films is examined.

Al_2O_3 was deposited via ALD at 300 °C using $Al(CH_3)_3$ and H_2O. 200 nm, 600 nm, and 1000 nm thick Al_2O_3 films were deposited on polished 305 stainless steel substrates. Prior to deposition, stainless steel substrates were cleaned using one of three methods: i) sonication in acetone, isopropyl alcohol and deionized water (AID), ii) AID followed by argon plasma treatment, iii) AID followed by oxygen plasma treatment. Nanowear, nanoscratch, and nanoindentation testing were performed using a Hysitron UBI-1 nanomechanical test system. A Berkovich diamond tip was used for nanoindentation testing to calculate the H and E at the interface of the ALD Al_2O_3 film with the 305 stainless steel substrate. A conical diamond tip was used to perform scratch

testing in order to quantify adhesion through measurement of delamination. The same conical diamond tip was also used for wear testing which characterizes wear resistance of a thin film.

Nanoscratch testing indicates excellent adhesion. The 200 nm Al_2O_3 films do not delaminate even when scratch depth into the sample extends to an average of 175 ± 0.1% of the film thickness. The 600 nm and 1000 nm Al_2O_3 films delaminate at an average of 102 ± 20% and 42.6 ± 3.8% of their respective film thicknesses. Reactive ion etching (RIE) treated samples also show less scratch resistance than untreated samples that only have the ALD Al_2O_3 coating. Nanoindentation exhibits a trend of H and E changing from literature ALD Al_2O_3 values to bulk stainless steel values with increasing penetration depth. Nanowear testing demonstrates that ALD Al_2O_3 films offer effective protection of the 305 stainless steel substrate, reducing wear by up to 74.1 ± 5% of the resulting values for wear depth of the bulk 305 SS samples. However, no distinguishable difference in mechanical properties or wear resistance was observed due to RIE treatment.

From the significant contribution that ALD Al_2O_3 provides for the improvement of wear resistance and scratch resistance, it may be concluded from this study that ALD Al_2O_3 exhibits strong potential for the improvement of orthopedic devices.

Key Words: atomic layer deposition, nanomechanical, atomic force microscopy
Corresponding e-mail address: nicolelbauer@gmail.com

Nanomechanical Characterization of Atomic Layer Deposition Coatings for Biomedical Applications

by

Nicole L. Bauer

A PROJECT

submitted to

Oregon State University

University Honors College

in partial fulfillment of
the requirements for the
degree of

Honors Baccalaureate of Science in Chemical Engineering (Honors Scholar)

Presented 22 April 2011
Commencement June 2011

Honors Baccalaureate of Science in Chemical Engineering project of Nicole L. Bauer presented on 22 April 2011.

APPROVED:

Co-mentor, representing Electrical Engineering and Computer Science

---

Committee Member, representing Electrical Engineering and Computer Science

---

Co-mentor, representing Biomaterials and Biomechanics

Committee Member, representing Biomaterials and Biomechanics

Dean, University Honors College

I understand that my project will become part of the permanent collection of Oregon State University, University Honors College. My signature below authorizes release of my project to any reader upon request.

Nicole L. Bauer, Author

Acknowledgements

<u>Oregon State University</u>

Dr. John Conley, Professor of Electrical Engineering and Computer Science

Co-mentor, provided oversight of sample synthesis procedures

Sean Smith, Ph.D Candidate of Materials Science

Assisted in sample preparation, specifically in atomic layer deposition

<u>Oregon Health & Science University</u>

Dr. John Mitchell, Associate Professor of Biomechanics and Biomaterials

Co-mentor, provided oversight of nanomechanical procedures

Dr. Mansen Wang, Postdoctorate Researcher of Biomechanics and Biomaterials

Assisted in the performance of additional nanomechanical tests

<u>With Funding and Assistance Provided by</u>

Semiconductor Research Scholars & Ellen Momsen

Hysitron Corporation

Table of Contents

I. Introduction..... 1

II. Background

Reactive Ion Etching..... 3
Atomic Layer Deposition..... 3
UBI-1 Nanomechanical Testing System..... 4
Nanoindentation..... 5
Overview of Scratch Testing..... 7
Overview of Wear Testing..... 8

II. Materials and Methods

Surface Preparation..... 9
Deposition of Al_2O_3..... 9
Nanoindentation Tests..... 10
Wear Tests..... 11
Scratch Tests..... 12

III. Results and Discussion

Nanoindentation Results..... 13
Scratch Test Results..... 17
Wear Test Results..... 21

IV. Conclusion..... 23

V. Bibliography..... 25

List of Figures

1. Atomic Layer Deposition Reaction Cycle..... 4

2. Load Displacement Curve..... 6

3. Nanoindentation Results.....13, 14, 15

4. Atomic Force Microscopy Scratch Image..... 18

5. Scratch Depth at Delamination..... 18

6. Scratch Depth at Peak Load..... 20

7. Wear Depth..... 21

Dedication

To Benjamin Huntington, whose brilliant mind and genuine

love lays the foundation of my endeavors.