IBAD Nanothick Bioceramic Incorporation on Metallic Implants for Bone Healing Enhancement. From Physico/Chemical Characterization to In-vivo Performance Evaluation

P.G. Coelho* and J.E. Lemons**

* University of Alabama at Birmingham, Materials Science and Engineering, Microgravity Solidification Laboratory, 1150 11th Ave south, Birmingham, AL, USA 35205, coelhp96@uab.edu
** University of Alabama at Birmingham School of Dentistry, Orthopedic Research Laboratory, 1919 7th Ave south., Birmingham, AL, USA 35205 jlemons@uab.edu

ABSTRACT

Preliminary data on in-vivo performance of nanothick bioceramic IBAD-coated metallic implants showed an increase in both osteoblastic activity and mechanical properties for bone surrounding these implants. The purpose of this study was to characterize a thin-film bioceramic coating obtained by Ion Beam Assisted Deposition (IBAD) on Ti-6Al-4V implants and evaluate its in-vivo performance in an animal model. Control (C) and IBAD-coated implants were analyzed by SEM, EDS, ion-milling + XPS for depth profiling, and thin-film XRD. 60 cylindrical implants were bilaterally placed in the tibias of 6 beagle dogs through sequenced surgical procedures and were biomechanically tested (torque) at 3 and 5 weeks in-vivo. No thin-film was detected by SEM, and the EDS spectra for IBAD coated implants showed Ca presence. XPS showed Ca, P, Si, C, and O at outer layers with variable Ca/P ratios as a function of depth. XRD spectra revealed an amorphous microstructure for IBAD implants. Biomechanical tests showed that IBAD-coated implants had superior fixation competence compared to control implants at 3 and 5 weeks in-vivo. According to the results, it was concluded that the nanothick surface coating enhanced the biological response of bone to implant, supporting opportunities for increased bone healing response in clinical practice.

Keywords: *IBAD, Thin-film, Bioceramic, Characterization, Biomechanical Testing.*

1 INTRODUCTION

Nanotechnology has strongly affected biomaterials science and engineering, once reduced condensed matter domains may deeply alter biomaterials' electronic/thermodynamic properties, and thus the response of living tissues to them.

Once the term osseointegration [1] (direct bone contact to titanium and its alloys) was first defined by a Swedish group, significant increases in orthopaedic/ dental surgical research and clinical trials occurred to investigate this phenomenon's underlying mechanisms. After the confirmation of highly successful treatment planning protocols, where metallic implants were surgically placed and an arbitrary healing time (3 to 6 months osseointegration time) was allowed before implant loading and function, significant interest have been devoted to metallic surgical implants' design modification, in an attempt to decrease healing periods, potentially benefiting patients and health care practitioners.

Among the changes that have been performed in order to enhance bone healing around implants, the addition of calcium- and phosphate- based materials as coatings have been performed by various methods [2-4], because these elements are the basic components of natural bone and have showed promising results in animal and clinical trials.

Despite its success in clinical trials, the commercially available Plasma Sprayed Hidroxyapatite (PSHA) bioceramic coatings have various process-inherent limitations, including coating thickness (~20-50µm which will remain without dissolving for long periods in-vivo [5-6], where bone never come to direct contact to the metallic substrate), multiphase microstructure (yielding variable dissolution/coating breakdown), and weak bonding between coating and substrate (mechanical interlock-metalloceramic link) [4,7-8].

In an attempt to improve on these circumstances, thin-film bioceramic coatings have been engineered for implant surfaces through processes like sol-gel deposition, pulsed laser deposition (PLD), ion beam assisted deposition (IBAD), and electrophoretic deposition [3]. These techniques often result in substantially thinner coating thicknesses compared to PSHA coatings [2]. Desirable features of thin-film coatings include controlled composition and thickness plus enhanced adhesion to the metallic substrate [2,4]. Controlled composition and thickness achievable through any of these processes influences coating dissolution in-vivo, providing osseoconductivity at early implantation times (coating tailored dissolution yielding exposure of the metallic substrate for bone anchorage), thereby influencing the bone-implant mechanical properties [8-10].

In-vivo studies [9-10] on bone response to Ca- and P-based IBAD processed thin-film coatings have demonstrated increases in osteoblatic activity at early

implantation times compared to control (non coated) implants. Other studies [10] have indicated higher bone-biomaterial interfacial shear strength values for IBAD coated implants versus non surface modified implants. These studies [9-10] also provided evidence that the IBAD processed thin-films were totally dissolved from the implant surface at early implantation times, supporting favorable conditions for implant fixation for subsequent functional loading, avoiding the bone-bioceramic and a bioceramic-substrate [5-6]interfacial transitions present in thick coated commercially available devices.

The purpose of this study was to physically/chemically characterize, and further investigate the in-vivo performance of IBAD processed nanothick bioceramic coatings in Ti-6Al-4V surgical implants through biomechanical (torque to failure) testing.

2 MATERIALS AND METHODS

The 4 mm in diameter and 10 mm in length implant rods with IBAD-coated and control (C) (grit blasted and acid-etched) surfaces were provided by the manufacturer (Bicon, Inc., Boston-MA).

2.1 Physico/Chemical Characterization

SEM and EDS- SEM and EDS assessment were performed at different locations throughout the implant surface at various magnifications.

XPS- The overall bulk surface composition survey was performed by XPS (160 eV, Kratos Axis 165 multitechnique) in various locations of the implant surface.

Depth Profiling- Chemical composition depth profiling was performed by ion-milling (4 keV Ar^+ ion sputter gun) the implant surfaces at 5 minutes (~5 nm/min rate for silica standard specimen) intervals followed by XPS (80 eV , Kratos Axis 165 multitechnique) evaluation.

Thin-film XRD- The implants were scanned on the thin-film x-ray diffractometer with starting scan angle of 30 degrees and the final 38 degrees.

2.2 In-vivo Surgical Model

The laboratory in-vivo model comprised 6 male beagle dogs (1.5 years of age average) in good health. These animals were obtained and followed during a 4-week housing period before the surgical interventions.

The surgical sites were the proximal tibiae, where 5 implants were placed on each posterior limb. The left and right limbs provided specimens that remained for 5 and 3 week periods respectively.

Following sterile procedures, the soft tissues were incised and manipulated for bone exposure and the implants were placed following the manufacturer's recommendation. The first implant was placed 2 cm from the joint capsule and the remaining devices placed 1 cm from each other along the bone (Figure 1). The site was closed by standard layer techniques utilizing 5-0 vycryl resorbable suture for the inner layers and 3-0 nylon suture for the outer layer.

2.3 Biomechanical Testing

After euthanization, the limbs were removed and the implants exposed by sharp dissection. Each implant received a screw on its exposed site and the tibiae were positioned in a torque testing device adapted to a universal testing machine (Figure 2). The implants were torqued to failure at a 0.05 in/min rate and the maximum force for torquing value recorded for each specimen. Analysis of variance was performed at a 95% level of significance.

Figure 1: Implants placed in proximal tibiae.

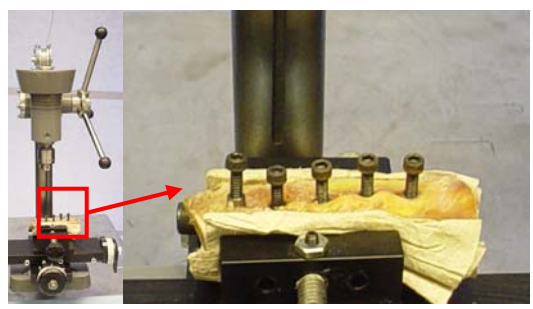

Figure 2: Specimen adaptation to torque testing apparatus.

3 RESULTS AND DISCUSSION

The SEM micrographs at 2000x magnification for the IBAD control surfaces are presented in Figure 3 along with their respective EDS and TFXRD spectra. Evaluation of the micrographs at 2000x revealed no evidence of a bioceramic layer on the implants surface. The control and IBAD surface topographies presented similar morphologies, consistent with sand blasted and acid etched surfaces. The EDS chemical analyses (Figure 3) showed Ti and Al were detected for control specimens and small amounts of Ca along with Ti and Al for IBAD-coated specimens.

The thin-film XRD spectra are presented in Figure 3 and revealed that the IBAD bioceramic thin coating presented an amorphous microstructure, once there was no evidence of HA peaks at 31 and 33 degrees 2 theta along

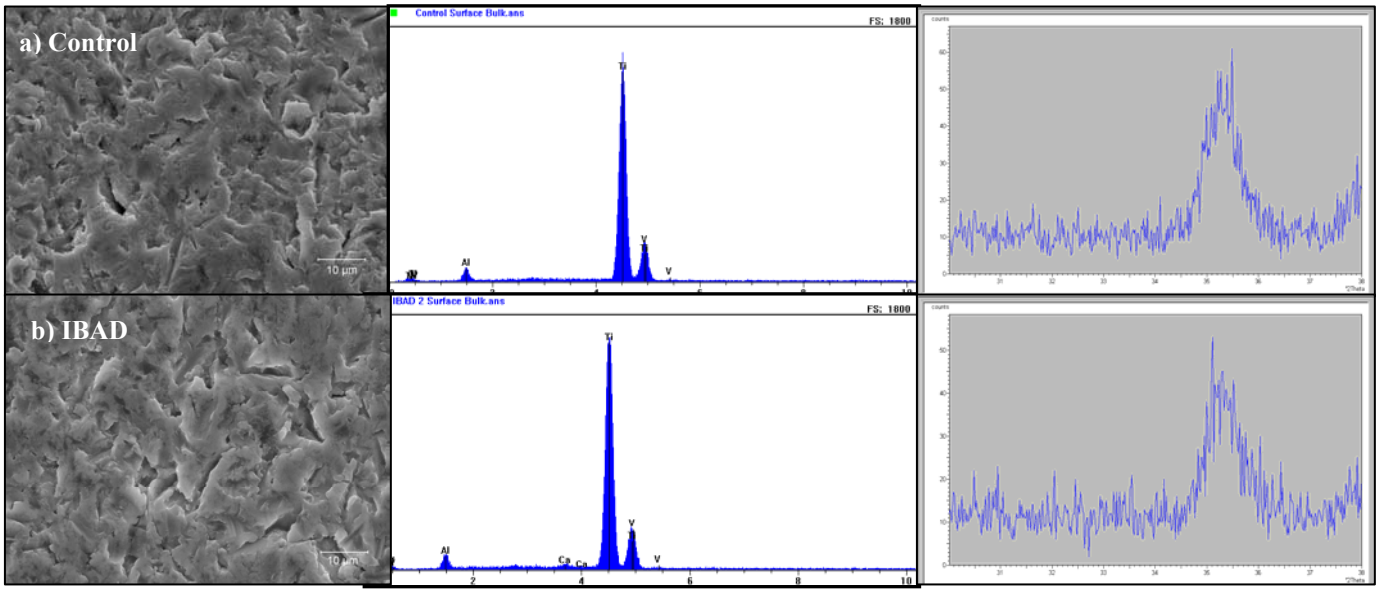

Figure 3: SEM, EDS, and TFXRD for Control (a) and IBAD-coated (b) implants.

with the presence of an alpha Ti peak at approximately 35.5 degrees 2 theta.

The XPS survey analyses revealed the presence of Ti, Si, O, C, and Al for control group implants and Ca, P, O, Si, and C for the IBAD implants. The presence of C and Si was due to surface adsoption of these elements. The absence of Ti and Al in the IBAD-coated specimens' spectra revealed that the thin-film was uniform along the implant surface. The Ca/P ratio (stoichiometry) depth profile is presented in Figure 4. The elemental composition profile revealed that the IBAD coating is in the nanothick range since Ti and Al were observed as early as 800 seconds of etch time.

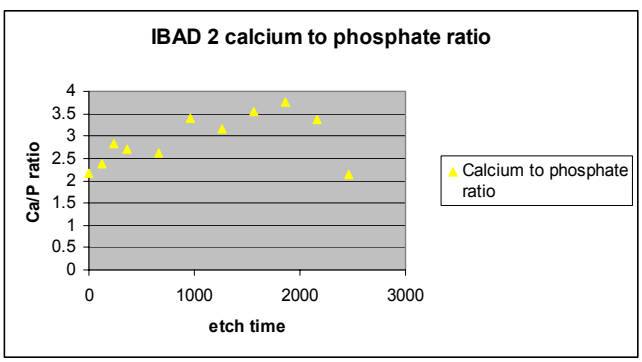

Figure 4: IBAD-coated implant Ca/P ratio depth profile

Evaluation of all analytical techniques as a group revealed that IBAD-coated implants were composed of amorphous Ca- and P- based bioceramic material in a nanometer range thickness. The two characteristics present for this bioceramic nanothick coatings are desirable to enhance the bone response to implants [2-4], and add the potential of total dissolution enabling direct bone anchorage to the metallic substrate due to low thickness and the high dissolution nature of amorphous bioceramic coatings.

Despite the variable stoichiometric characteristic as a function of coating thickness found for the IBAD-coated implants, these may be heat treated for crystalline HA obtention in the eventual need of altering this nanothick coating dissolution kinetics.

The ANOVA summary statistics for the torque force to failure test for both IBAD-coated and control groups at 3 and 5 weeks in-vivo periods are presented in Tables 1 and 2. The dependent variable was torque force to failure and the independent variables surface type and time in-vivo. These results showed that the range of maximum force values were similar for both times in-vivo and osseointegration [1] occurred for both implant surfaces at early implantation times, revealing that both surfaces are biocompatible and osseoconductive.

Source		
Main Effects	F Ratio	P- Value
a- Surface	42.83	0.0000
b- Time in-vivo	1.08	0.3170

Table 1- Independent variables F- and P- values revealed a surface effect on torque to failure force.

Results in Table 1 indicate that surface treatment had a significant effect ($P < 0.05$) on the dependent variable (Max Force), and that time in-vivo did not ($P > 0.31$). The 95% confidence intervals presented in Table 2 and Figure 5 show that the IBAD coated implants presented significantly higher Max Force (lbf) values for interface failure, revealing a coating effect [4-5,9-10] on osseointegration kinetics at times as early as 3 weeks in-vivo, supporting favorable conditions (early healing and fixation through bone apposition) for early implant loading and also revealing the IBAD processing of bioceramic coatings as an alternative for coating manufacturing [2-4, 9-10].

Level	Mean	All values in lbf	
Grand Mean	8.275		
Surface		Lower Limit	Upper Limit
Control	6.4	5.592	7.213
IBAD	10.143	9.22	11.067
Time in-vivo			
3 weeks	7.98	7.121	8.834
5 weeks	8.57	7.697	9.443

Table 2: 95% Confidence intervals for the different surfaces and times in-vivo.

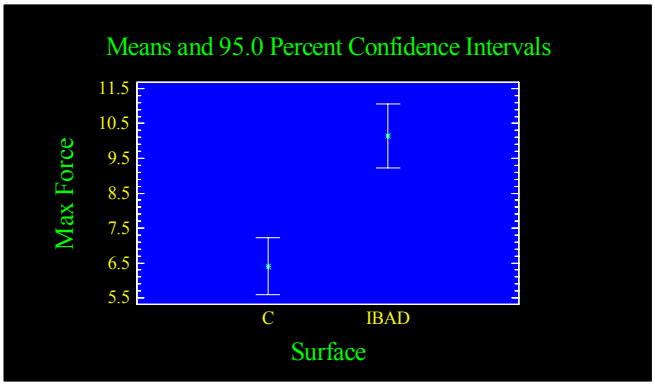

Figure 5: 95% confidence intervals for IBAD and Control Groups revealed higher fixation competence for the IBAD-coated implants. Max Force in lbf.

4 CONCLUSION

According to the in-vitro and in-vivo results obtained in this study, it can be concluded that nanothick bioceramic IBAD-coated titanium alloy implants may be regarded as an alternative to currently commercially available thick bioceramic coatings.

The characterization aspect of this study showed that the bioceramic amorphous nanothick film has morphology and composition that not only favors bone close contact and subsequent anchorage directly to the metallic substrate, but also enhances the healing of bone around coated implants (as per biomechanical testing results, where a higher degree of fixation was obtained for IBAD-coated implants).

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