Curvature Load on the Hydroxyapatite Coated Hip Implant Surface

Takeshi Otsuka*1, Shinji Nakagaki*2, and Tetsuya Nakamura*3

Abstract

The purpose of this research is to discuss the effect of Simulated body fluid immersion on the adhesive strength for hydroxyapatite (HAp) coating using the four-point bending test with a slit coated specimen. During testing, AE signals were recorded and delamination behavior from the edges of HAp coating was observed. In air condition specimens, stresses when AE signals were firstly detected were in a narrow range from 120 MPa to 130 MPa, which can demonstrate the effectiveness of AE signals in order to detect an initiation of delaminations of HAp coatings. However, after SBF immersion, a stress when AE detected shifted stronger region, which indicated AE characteristics during immersion would change, which requires the measurement of 4points-bending testing to tune the AE measurement conditions in order to successfully detect the initiations of HAp delaminations.

Key Words: Acoustic Emission, Adhesive Strength, Delamination, Four-point Bending Test

1. Introduction

As population ages and physical conditions become to deteriorate, biomimetic implants in different parts of the body have become reliable for in order to continue a safe lifestyle. Implant materials require high strength and resistance to the corrosive environment inside the body. Such is the case for the hip implant alloy Ti-6Al-4V, used as a base material for this investigation, which has been coated with a Hydroxyapatite layer (HAp: Ca(_10)(PO_4)_6(OH)_2) by plasma spray. The HAp coating resembles human bone and attaches with the bone. However, as adhesion strengths between the titanium alloy and the HAp coating are considered low and then a delamination of HAp coatings thought to be occurred when subjected to load[1]. According to the ASTM Standard Test Method for Tension Testing of Calcium Phosphate and Metallic Coatings, tensile loads are used to evaluate the adhesive strength of coating layers[2]. In the case of tensile testing, the results should be affected by adhesion conditions between HAp coatings with adhesives, which make the result more difficult to be interpreted. On the other cases of 4-points or 3-points bending test, the fracture mode is not simple delamination but the mixing mode of perpendicular cracking with subsequent delamination. In the conventional bending test cases, it is difficult to separate delamination strength from cracking strength. We then consider the feasibility of 4-points bending test with slit specimens to detect delamination strength from the edges of the HAp coatings[2,3]. However, the effect of edge angles on delamination strength should be considered because dissolution effect by SBF will also affect the shapes of the edges.

The present study aims at revealing the effects of edge angle's variations on delamination behaviors of plasma-sprayed HAp coating. We prepared two types of coatings; (A) plasma-sprayed HAp coating (B) plasma-sprayed Ti / HAp mixed

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coatings. Specimens were immersed in SBF for various periods and then discussed the effects of dissolution on delamination initiation behaviors.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Mechanical properties of Ti-6Al-4V alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2% Proof Strength (MPa)</td>
<td>Tensile Strength (MPa)</td>
</tr>
<tr>
<td>946</td>
<td>1016</td>
</tr>
</tbody>
</table>

![Microstructure of Ti-6Al-4V](image1)

![Schematic view of specimen during 4 point bending test](image4)

**Fig. 1** Microstructure of Ti-6Al-4V. **Fig. 4** Schematic view of specimen during 4 point bending test.

### 2. Experimental Procedure

#### 2.1 Testing Materials

A substrate material used in 4-points bending test is Ti-6Al-4V with dimensions 50x10x3 [mm]. Table 1 shows the mechanical properties of the Titanium alloy and Fig. 1 illustrates its microstructure. HAp powders used for the coating were HAp-100 (Taihei Chemical Industry) which were sintered at a temperature of 1473 K x 3.6ks in an electric furnace (Isuzu MRH-32UHS). After the sintering, the powders were crashed in a ball mill and filtered to achieve a fine grain size less than 90 μm.

Surfaces of substrates were grit-blasted in advance. Blast process was conducted by Fuji Pneumatic Blaster equipment at a constant pressure of 5 MPa with alumina particles (#30). In order to specify the delamination initiation point, a masking bar of 3mm of width was utilized as shown in Figure 2. HAp plasma spray was performed by the SulzerMetco 9MB (68V, 500A, at 140mm of distance) for 20 passes. For this sets of specimens the average thickness of HAp coating was 46 μm with a standard deviation of 7.0μm for the In Air condition group and an average of 47μm with a standard deviation of 5.3μm for the specimens submerged 1 week in the SBF. These values were obtained by measuring 10 points from the edge to a distance of 300μm for each specimen.

#### 2.2 Polishing Method of the Lateral Edge of the Plasma Sprayed Coating Specimen

The side surfaces of the specimens were polished to observe a clearer interface between HAp coating with Ti alloys substrates. The polishing process was made using Struers Automatic Polishing Machine starting with SiC paper #800 and MD-Dac with DP Suspension 9, 3, 1 μm. A force of 10 N was applied in each process to avoid damaging on the HAp coating.

#### 2.3 Simulated Body Fluid Preparation and Maintenance.

SBF was prepared according to the specifications of a recipe and exchanged every 2 days to maintain Ca levels as closest to in vivo environment as possible.

#### 2.4 Adhesion Strength Evaluation Test Method

The testing equipments used were the Shimadzu Servopulser machine (10kN) and the 4830 Shimadzu Controller. The test was conducted under displacement control at 0.004 mm/s. A strain gage was attached on the compressive side of Ti alloy substrates at its center. Two AE sensors were placed on the HAp coating surface with enough grease as show in Figure 4. The usage of 2 AE sensors is to reduce noises before a preamplifier. To limit the signals coming from only one edge, Strain Gage Cement CC-33A was applied on the remaining 3 edges. The parameters used to determine the adhesion strength were strain, stress and AE Signals along with the FFT Analysis.
After testing, the defined edge was observed in the SEM (JSM-6306A) to detect delamination initiations at the edges of HAp coating interface.

**Fig. 5 Testing Network**

<table>
<thead>
<tr>
<th>Machine Name</th>
<th>Model Number</th>
<th>Performance</th>
</tr>
</thead>
<tbody>
<tr>
<td>AE Sensor</td>
<td>AE-900M TYPE1</td>
<td>300kHz - 2MHz</td>
</tr>
<tr>
<td>AE Signals Module</td>
<td>As-712</td>
<td></td>
</tr>
<tr>
<td>Pre-Amplifier</td>
<td>AE 912</td>
<td>50kHz - 2MHz -40dB, fixed gain</td>
</tr>
<tr>
<td>Discriminator</td>
<td>AE9922</td>
<td>1kHz - 2MHz -40dB, gain</td>
</tr>
<tr>
<td>Digital Oscilloscope</td>
<td>DS-5324</td>
<td>200MHz - 2GS/s</td>
</tr>
</tbody>
</table>

### 3. Experimental Results and Discussions

#### 3.1 Shape of Sprayed Coating Edge

Figures 6 and 7 show an example of the edge formation of the HAp coating obtained with the masking. The objective of the masking is to get steeper angles on both lateral view and top view in relation with the HAp coating on the specimen. Table 3 shows the comparison between the specimens that were not submerged in the SBF and the specimens which were in the SBF for 1 week. Mean value of the edge angle as well as the standard deviation are expressed for each case. The values for the angle measurement were recorded before testing and before SBF submersion. It is considered that the closer the angle is to 90°, adhesion strength and delamination evaluations are more accurate.

#### 3.2 Adhesion Strength Evaluation Test

Figures 8 and 9 show the Stress-Strain relation curves and the AE signals related to HAp frequency for the two sets of specimens; In Air condition and 1 week SBF immersion. The specimens In Air, HAp first related frequency signals were registered at about 120MPa – 130MPa as shown in Fig. 8. Mean and Standard deviation of bending strength on three specimens are 124.1 MPa and 6.0 MPa, respectively. In the case of SBF immersion for 1 week, the HAp coating layer would be weakened. However, the stress range when the first HAp related frequency signals were detected became higher compared with those from no immersion case. The results indicate that stress concentrations at the edge of coatings are critical factors in determining the level of delamination strength. Furthermore, the variations in edge angles are more affective in delamination strength compared with the dissolution effects by 1 week immersion in SBF. In the case of 63.5degree, its singularity coefficient in the pair of HAp with Ti substrates is 0.306 whereas the singularity coefficient is 0.274 in the cases of 54.8 degree by Bogy’s eigen equation. The slight differences in the singularity coefficients, which lead to small differences in elastic singular stress distribution, may attribute to little changes in the values of energy release rates. Further discussion is needed to apply energy release rate G whether the current result can be explained by the changes in the values of G. Another possibility is that the large scatter in AE signal behaviour for the 1 week immersion case could be related to the changes in acoustic properties of HAp coatings such as increasing in porosity or precipitation.
of amorphous Calcium Phosphate. Further investigation is necessary to determine the correlations between frequency spectrums of AE signals with delamination behaviours after long-term immersion.

![Fig. 6 Example of an edge's angle from the side view.](image)

![Fig. 7 Formation of edge top view.](image)

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Comparison of edge angles of the two sets of specimens.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Edge Angles</td>
<td>Mean Value</td>
</tr>
<tr>
<td>In Air</td>
<td>63.5</td>
</tr>
<tr>
<td>1 week SBF</td>
<td>54.8</td>
</tr>
</tbody>
</table>

![Fig. 8 σ-ε curve and AE signals in Air.](image)

![Fig. 9 σ-ε curve and AE signals after 1 week SBF immersion.](image)

**3.3 FFT Analysis**

Figures 10 and 11 display the FFT Analysis realized on the first HAp related signal over 2V registered for the In Air condition and the 1 week SBF groups. Relating to previous investigations, the frequencies of both signals which represent an alteration of the HAp coating are in the range of 400kHz – 600kHz or in the region around 800kHz. For both cases, it can be clearly observed how the wave formations correspond to the regions of cracking and delamination of the HAp coating from the substrate of the Ti alloy.

**3.4 SEM observations on interfacial delaminations after testing**

After conducting the test, observations by using SEM were conducted in order to identify a delamination initiation or cracking of the HAp coating. Figures 12 and 13 demonstrate the HAp coating interface before and after testing for the In Air case. Figure 12 presents no significant separation in between the substrate alloy and the HAp coating. After conducting the 4-points bending test for the same specimen, a slight shadow in between the substrate and the HAp coating suggest the initiation of delamination occurring. This image can be matched along the recorded HAp signals as evidence of the delamination of the HAp coating from the specimen during the test.
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Fig. FFT data of a typical AE signal for in Air

Fig.11 FFT data of a typical AE signal for 1 week SBF

Fig. 12 Surface features of HAp coating before tests

Fig.13 SEM image of delamination initiation at an interface

4. Summary

Throughout this investigation, HAp delamination initiation detection was successfully obtained by using AE signals and conducting the FFT analysis when the specimens were kept in air. After SBF immersion, AE generation behaviours were changed, which make the detecting of initiation points more complicated. The cause of changes in AE generation features are ye to be fully understood. Further experiment is necessary to reveal the effects of AE characteristics by SBF immersion separately from the effect of changes in morphology during SBF immersion.

The results on mixed Ti/HAp coatings as well as the 30 days SBF immersion will be added in the presentation in order to discuss the effect of SBF immersion on the changes in their strength or their edge shapes.

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