Optimal Condition of Hydroxyapatite Powder Plasma Spray on Ti6Al4V Alloy for Implant Applications

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Abstract Optimal conditions for HA plasma spray-coating on Ti6Al4V alloy were investigated in order to obtain enhanced bone-bonding ability with Ti6Al4V alloy. The properties of plasma spray coated film were analyzed by SEM, XRD, surface roughness measurement, and adhesion strength test because the film’s transformed phase and crystallinity were known to be influential to bone-bonding ability with Ti6Al4V alloy. The films were formed by a plasma spray coating technique with various combinations of plasma power, spray distance, and auxiliary He gas pressure. The film properties were analyzed in order to determine the optimal spray coating parameters with which we will be able to achieve enhanced bone-bonding ability with Ti6Al4V alloy. The most influential coating parameter was found to be the plasma spray distance to the specimen from the spray gun nozzle. Additionally, it was observed that a relatively higher film crystallinity can be obtained with lower auxiliary gas pressure. Moderate adhesion strength can be achievable at minimal plasma power. That is, adhesion strength is minimally dependent on the plasma power. The combination of shorter spray distance, lower auxiliary gas pressure, and moderate spray power can be recommended as the optimal spray conditions. In this study, optimal plasma spray coated films were formed with spray distance of 70 mm, plasma current of 800 A, and auxiliary gas pressure of 60 psi.

Key words plasma spray coating, hydroxyapatite, Ti6Al4V, implant.

1. Introduction

Titanium and some of its alloy (e.g. Ti6Al4V) are widely used as orthopaedic and dental implant materials due to their low elastic modulus, good biocompatibility and corrosion durability. However, bone does not bond directly to these materials. In order to enhance the bone-bonding ability, titanium and its alloys are often coated with HA (Hydroxyapatite) powder.

A plasma-sprayed coating of HA powder on Ti6Al4V alloy gets popular among various coating methods.1-5) It is due to fast bony adaptation, absence of fibrous tissue seams, strong implant-bone attachment, a reduced healing time, and inhibition of ion release etc. Thus, it can easily replace PMMA (Poly Methyl Metacrylate) cement fixation technique, which has been known to have deficiency such as stem release due to polymer degradation and mechanical failure.

In this study, an optimal spray-coating parameters were investigated and the coated film properties were examined. Critical parameters such as plasma current, spray distance and auxiliary gas (He) pressure were selected to determine an optimal recipe for spray coating.

2. Experimental Procedure

During plasma spray coating, HA powder (amorphous phase) can not be avoided to be exposed to an extreme high temperature and a rapid cooling down on a substrate surface, which will cause transformation into one of the crystalline phases. Those transformed phase and crystallinity were known to be influenced especially by the plasma spray processing conditions of plasma current, spray distance and auxiliary gas pressure. Thus, in this study, various

Table 1. Spraying parameters for HA coating.

<table>
<thead>
<tr>
<th>Current</th>
<th>Aux gas pressure 60 psi</th>
<th>80 psi</th>
<th>100 psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>800 A</td>
<td>A1, B1, C1</td>
<td>A2, B2, C2</td>
<td>A3, B3, C3</td>
</tr>
<tr>
<td>850 A</td>
<td>A4, B4, C4</td>
<td>A5, B5, C5</td>
<td>A6, B6, C6</td>
</tr>
<tr>
<td>900 A</td>
<td>A7, B7, C7</td>
<td>A8, B8, B9</td>
<td>A9, B9, C9</td>
</tr>
</tbody>
</table>

standoff distance
A: 70 mm, B: 85 mm, C:100 mm

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processing parameters were prepared and labeled as described in Table 1.

Amdry 6021 HA powder was used and its size was distributed to be 45-160 µm, as shown in Fig. 1. A substrate was Ti6Al4V with 10 mm in diameter and 5 mm in thickness. In order to increase adhesion strength, as a pretreatment, Ti6Al4V substrate surface was blasted to 6 µm (Ra) surface roughness with alumina grit (#24 mesh) with 5 kg/cm² air pressure, and 30-40 mm distance at 90° spraying angle.

The other plasma spray coating parameters were fixed as 50 psi of Ar arc gas pressure, 3 rpm of a hopper RPM, and 7.3 mm of a nozzle size respectively. The crystallinity of HA before and after coating was examined using scanning electron microscope (SEM) and X-Ray Diffraction (XRD) technique. (No peak broadening around 2q = 30°(2θ) was observed due to HA powder amorphous phase). The bond strength between the coating and substrate disc was measured five times in accordance with ASTM C633-01, the standard test method for the adhesive or cohesive strength of thermal spray coatings. A glue between HA coated specimens was Aremco- Bond®631.

### 3. Results and Discussion

#### 3.1 Comparison of crystallinity of HA with processing parameters

Fig. 2 shows XRD patterns of HA after plasma spraying with different standoff distance. A1, B1 and C1 specimens showed the highest crystallinity as shown in Fig. 2a and 2b.

It showed that the longer spray distance, the more CaO created and the less crystallinity of HA coated film as shown in Fig. 3.

Particularly, A1 specimen (the shortest spray distance) showed almost zero CaO phase creation and the highest crystallinity. It can be explained by that as the longer spray distance, the longer HA powder will be exposed in a liquid phase, which will accelerate TCP (tricalcium phosphate) and TeCP (tetracalcium phosphate) phase formation which is not desirable.

Fig. 4 shows XRD spectra for “A” specimens which have the shortest spray distance with various combination

![Fig. 3. The ratio of ICaO and IHA.](image)

![Fig. 2. (a) XRD patterns of hydroxyapatite after plasma spraying at incremental standoff distance and (b) XRD intensity of the principal HA peak.](image)
of current and auxiliary He gas pressure. As shown in Fig. 4 the higher spray current and auxiliary He gas pressure, the less crystallinity and non-desirable phase transition from HA phase occurred.

As auxiliary He gas pressure increases, HA powder spray speed increases accordingly. Thin lamellar structure was formed because the molten powder can spread more widely due to an increase of spray speed. Since thin coating layer have the higher cooling rate than a thicker one, OAP (oxyapatite) and amorphous phase formation, which require the higher driving force than one for HA phase, can be accelerated. A1 showed the less amorphorization and the less HA decomposition than A3 or A7. As shown in Fig. 5, coating layers close to a substrate has the thinner lamellar structure than those close to the top surface.

Amorphization occurs by the higher cooling rate due to quick absorption of the heat from HA powder to a substrate when molten powder strikes a substrate. HA recrystallization and crystallization in coated films can be increased by lowering the cooling rate due to its low temperature difference.

### 3.2 Adhesion strength of coated HA film

Fig. 6 shows the adhesion strength test results of that specimens.

The adhesion strength of the HA coated specimens was around 13–14 MPa except A3. From the result that A4, A5, A6 specimens have higher adhesion strengths than A1, A2, A3 specimens, it can be concluded that the higher spray power, the higher adhesion strength. The reason why
A3 specimen showed the lowest adhesion strength than the others can be explained by cracks (shown in Fig. 5) between the substrate and coating layers.

4. Conclusion

Most effective and influential condition on plasma spray coating was a plasma spray distance ("A" specimen) observed by XRD analysis. The optimal spray distance was 70 mm. It was observed that the higher a spray current and the higher an auxiliary He gas pressure, the less crystallinity and the higher non-desirable phase transition from HA phase occurred. As for the adhesion strength, it was found that minimal spray power is enough to obtain good adhesion strength, even though the adhesion strength is increased with the spray power. Considering all the test results, the optimal plasma spray coated films were formed with spray distance of 70 mm, plasma current of 800 A, and auxiliary gas pressure of 60 psi.

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References