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# Implant Surface Modification Using Laser Guided Coatings: In Vitro Comparison of Mechanical Properties

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**Keywords**
Hydroxyapatite; pulsed laser deposition; plasma spraying; bond strength.

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*Previously presented at the 82nd Annual IADR Meeting, Implant Prosthodontic Research Session, at Honolulu, HI, March 10-13, 2004.*

Accepted February 24, 2007

doi: 10.1111/j.1532-849X.2008.00307.x

## Abstract

**Purpose:** Plasma-sprayed hydroxyapatite (HA)-coated implants show failures along the coating–substrate interface due to poor bond strength. We analyzed HA coatings obtained by pulsed laser deposition (PLD) and compared them to commercially used plasma-sprayed coatings with respect to their bond strength to titanium alloy (Ti-6Al-4V), as well as surface roughness alterations produced by each of the two deposition methods.
**Materials and Methods:** Twelve titanium alloy disks were plasma-sprayed under commercial implant coating conditions, and 24 titanium alloy disks were coated using PLD. All coatings were characterized by the presence of the different calcium phosphate (CaP) phases. The plasma-sprayed coatings ($n = 12$) were predominantly HA, and the pulsed laser-deposited coatings were hydroxydyapatite ($n = 12$) and HA coating with a tetra calcium phosphate (TTCP) phase ($n = 12$). The surface roughness was analyzed before and after the coating processes to assess roughness changes to the surface by the coatings. The adhesive bond strengths of these coatings to the substrate titanium alloy was tested and compared. Scheffé's test was used to analyze the statistical significance of the data.
**Results:** The surface roughness alteration following PLD was a decrease of 0.2 $\mu$m, whereas following plasma spraying the decrease was 1.0 $\mu$m. Bond strengths were as follows [mean (SD) in MPa]: pulsed laser-deposited HA coatings: 68.3 (17.8); pulsed laser-deposited HA with tetra-CaP: 55.2 (21.1); plasma-sprayed HA 17.0 (2.8). The multivariate Scheffé's test revealed that HA coatings obtained by PLD had significantly increased bond strengths compared with the plasma sprayed ones ($p \leq 0.05$).
**Conclusions:** HA coatings obtained by PLD showed greater adherence to titanium alloy. PLD offers an alternative method to produce thinner coatings with better adherence properties, along with precise control over the deposition process.

Calcium phosphate (CaP) ceramics have been examined as materials for making biomedical implants for almost a century. Hydroxyapatite (HA), which is the main mineral in natural bone, shows the most desirable bone response among bioactive materials.[1] The stability of HA coatings, along with their chemical bonding to bone, showed their potential for use in clinical applications.[2] These coatings typically exhibit faster bony adaptation and show an absence of fibrous tissue seams along the surface of the coating. These coatings provide a firmer implant attachment by the formation of a chemical bond with bone and reduce healing time by an earlier integration with bone.[1] One of the main advantages of the coating is that it inhibits the release of metal ions, which is not desirable for osseointegration as it will lead to bone loss around the implant. On analyzing the literature, it has been shown that HA coatings can promote earlier and stronger fixation of the implant but exhibit a durability that can be related to the coating quality.[3] HA-coated implants have been shown to be more successful than non-HA coated implants in type 3 and type 4 bone.[4]

In order to make informed decisions regarding HA-coated products, clinicians need to familiarize themselves with some critical variables of HA coatings, such as crystallinity, purity, thickness, bond strength, and dissolution rate.[5,6] Endosseous dental implants are available with various surface characteristics and modifications. One of these is surface roughness, which has been shown to improve bone-to-implant contact. The surface roughening process increases the surface area of the implant greatly, thereby contributing to greater bone-to-implant contact. HA-coated implants, by virtue of their processing, are rough-surfaced and thereby increase surface area. Due to their ability to show chemical bonding with bone, they exhibit a more rapid decrease in micromobility than do titanium plasma-sprayed implants of identical geometry. HA coatings also tend to accelerate the initial rate of osseointegration.[7]

Currently, the commercial practice for applying HA coating for implants is by plasma spraying. For a plasma-sprayed coating to exhibit reasonable adhesive bone strength, the surface of the alloy needs to be roughened. This still does not address a major problem: the fracture of the coating that occurs in and along the interface of the coating during implant placement or during load-bearing situations. This is possibly due to the thickness, porous nature, and a nonuniform crystallinity of the coating with poor adherence of the coating to the substrate.[8] As the plasma-spraying method cannot produce coatings within a few microns range, attempts to improve the coating stability by heat treatments have resulted in delamination of the entire plasma-sprayed coating at the time of implantation. Studies have shown that the mechanical adhesion of the CaP coatings to the metal substrate is dependent upon the thickness and phases of the coating, along with the adhesive bonding between the alloy and the coating. Thin coatings have shown promise for controlling these properties, and some of the methods available to make these thin coatings include magnetron sputtering, pulsed laser deposition (PLD), ion beam-assisted deposition, electrophoresis, surface-induced mineralization, bio-mimetic deposition, and sol-gel processing.[9]

Deposition of HA coatings usually shows the presence of other CaP phases along with that of HA. The most common CaP phases that form during the deposition of HA are tetra calcium phosphate (TTCP) and tricalcium phosphate ($\alpha$ and $\beta$). It has been shown that the success of HA coatings used to accelerate initial bone growth on dental implants can vary depending on the CaP phases present in the coating.[10]

PLD is a versatile experimental method that finds use in a diverse range of materials and in wide areas of thin film deposition and multilayer research.[11] The underlying chemistry and physics are still not very well understood. Pure crystalline HA films with thicknesses of less than 10 $\mu$m have been deposited on titanium metal and alloy using the PLD technique.[12] Use of a PLD system is a recent development in the field of HA coatings and shows promise for achieving coatings of uniform thickness and improved control in the presence and absence of other phases. The PLD coating has previously shown acceptable adhesive bond strengths on smooth- and rough-surfaced alloys.[13] In this study, the PLD method was investigated as an alternative coating technique. The aim of this study was to compare the surface roughness alterations produced by the two methods of coating and to test the adhesive bond strengths of HA coatings obtained by PLD and plasma spraying.

## Materials and methods

### Substrate preparation

A total of 36 titanium alloy (Ti6Al4V) disks (1-cm diameter, 2-mm thick) were used as substrates for coating the HA. The disks were categorized into two groups; one group of specimens (n = 12) was smooth-surfaced and the other group (n = 24) was surface roughened under commercial implant coating conditions. The smooth-surfaced specimens were coated by the PLD method only. Within the rough-surfaced specimens, 12 received coatings by plasma spraying and 12 by PLD.

The 12 smooth-surfaced specimens were prepared by wet-polishing the alloy disks with silicon carbide papers with grits of 180, 600, and 1200 and polycrystalline diamond suspensions of 6 and 1 $\mu$m in a sequential manner. The smooth-surfaced specimens were then cleaned with acetone, methanol, and deionized water in an ultrasonic bath. The surface roughening of the 24 specimens was performed by Biocoat, Inc. (Southfield, MI), where the surfaces were grit-blasted and acid-etched.

### Profilometry

Surface roughness analysis of the specimens was done before and after obtaining the coatings with both the PLD and the plasma spraying process. All 24 rough-surfaced and 12 smooth-surfaced specimens were analyzed for roughness average (Ra) values before the coatings were obtained. The Ra values were then obtained again after the coating process was done, to verify changes in the roughness induced by the coating method.

The surface roughness analysis was done by using a diamond tip surface analyzing profilometer. Ra values of the titanium alloy substrates and the coatings were obtained using a stylus profilometer. Each specimen received five different surveys in different areas with the scan length being approximately 8 mm. The Ra was tabulated from the scan graphs.

### Plasma spraying process

Twelve rough-surfaced specimens were coated with HA by plasma spraying. The plasma spraying process is a method where molten HA powder is injected into a very high temperature plasma flame, where it is rapidly heated and sprayed onto the surface of the alloy to provide a coating. The hot molten powder impacts on the alloy surface and cools rapidly, forming the coating.

### Pulsed laser deposition

Twenty-four specimens (12 rough-surfaced, 12 smooth-surfaced) were used to obtain the PLD coating. In this method (Fig 1), the authors employed the use of a KrF excimer laser (248 nm), with an energy density of 4-10 J/cm$^2$ and a repetition rate of 30 Hz while the substrate temperature was kept between 625 and 715°C. A deflecting mirror was used to guide the laser into a controlled atmosphere chamber where it ablated the HA target to generate a plume of atoms, ions, and particles. These particles then coalesced and deposited themselves onto the surface of the heated, rotating alloy in an argon/water atmosphere, thereby forming the coating.

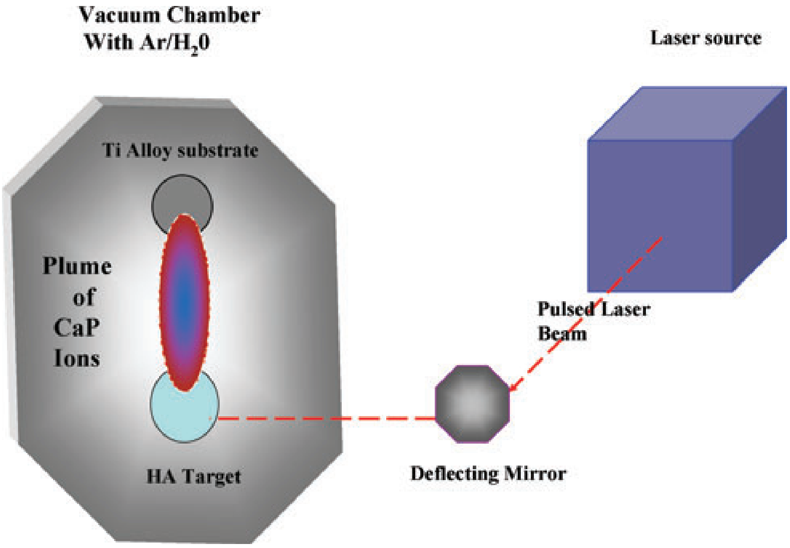

**Figure 1** Pulsed laser deposition of hydroxyapatite coating over titanium alloy.

## X-ray diffraction

An X-ray diffractometer was used to characterize the as-deposited PLD CaP coatings along with the plasma-sprayed coatings. Parameters of the X-ray diffraction were 40 mA and 45 kV using a copper target to achieve a wavelength of 1.540 nm. The "2 theta" scan range began at 25° and ended at 45° with a step size of 0.050° and a dwell time of 5 seconds, leading to 500 data points per specimen in a continuous fashion. The collimator angle was 0.27°, which is normally used for thin film diffraction. The coatings obtained by PLD were grouped based on results of X-ray diffraction (XRD) analysis of crystallinity: high crystallinity HA and lower crystallinity HA with TTCP.

## Bond strength testing

The coatings were tested to determine their bond strength using a Z-axis pull test. A Sebastian Five adhesion testing apparatus (Quad Group, Spokane, WA) was used for testing the tensile bond strength of these coatings. This system has a maximum load of 100 Kg and maximum pull rate of 9 Kg/sec. The test was performed using nail-like aluminum pull studs, which have a coating of epoxy resin on the head. The diameter of the head of these pull studs was 0.106 inches. These pull studs were clipped onto the specimen using specific mount clips to hold these stud heads attached to the sample. The specimen with studs and the mount clips were then placed into a hot air oven for 1 hour at a temperature of 150°C followed by 60-minute bench cooling to cure the epoxy-coating bond. Testing was started with two uncoated titanium alloy disks (1 smooth, 1 rough) to assess the similarity in testing conditions and to verify the strength of the epoxy resin when bonded directly to the alloy. Scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) was used to assess the mode of fracture in the following regions: within the coating, within the epoxy,

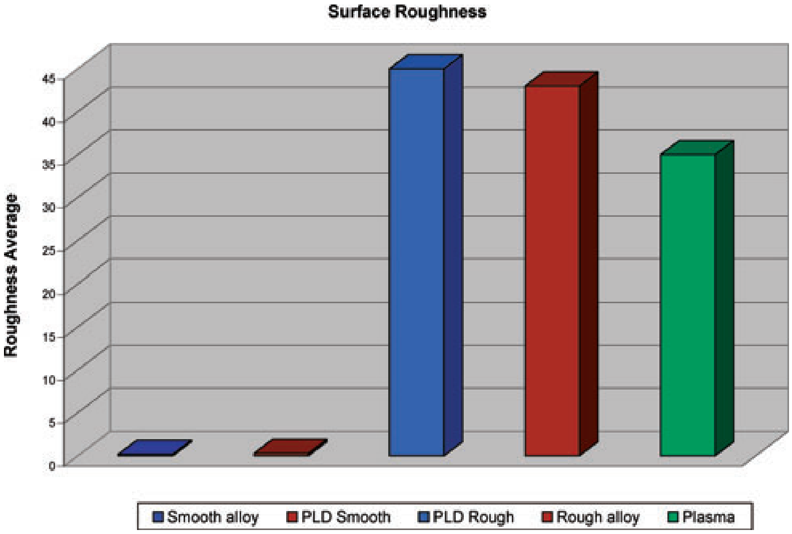

**Figure 2** Graphical representation of roughness distribution in $\mu$m.

**Table 1** Profilometer results ($\mu$m)

| Specimen type | n | Mean roughness (Ra) | Standard deviation |
|---|---|---|---|
| Smooth polished alloy | 12 | 2.0 | 1.0 |
| PLD coating on smooth polished alloy | 12 | 3.0 | 1.0 |
| Rough-surfaced alloy | 24 | 43.0 | 2.65 |
| PLD coating on rough alloy | 12 | 45.3 | 1.15 |
| Plasma-sprayed coating on rough alloy | 12 | 35.3 | 5.1 |

the coating–substrate interface, the coating–epoxy interface, and between the epoxy and the pull stud. The load rate used in these tests was 5 kg/sec, with the stress rate being approximately 68.95 MPa. A Scheffé test and ANOVA were done to analyze the statistical significance at $p \leq 0.05$.

### SEM/EDS

The as-deposited PLD and plasma-sprayed specimens were analyzed for surface morphology with SEM. SEM with EDS was used to identify the base elements present in the coating. EDS is a method by which the elements present in any material are identified. SEM analysis was performed after the bond strength tests to assess the nature of the bond failure. EDS was used to assess if the fracture completely delaminated the coatings without leaving any residue, or if fracture occurred between the layers. If the coating had any residue left behind in the fractured area, it would have been identified as calcium or phosphorus, which are the base elements in the CaP coatings.

## Results

### Characterization of the coatings

The coatings obtained by the PLD process and the plasma-spraying process were characterized using EDS, SEM, XRD, and profilometry. Alteration in surface roughness of the alloys was evaluated to assess the roughness changes after deposition of the coatings. The distribution of the roughness averages (Ra) is shown in Table 1 and is displayed graphically in Figure 2. The smooth-surfaced alloy samples without any coating showed Ra values of about 2.0 $\mu$m, whereas after obtaining the coating they increased in roughness values to 3.0 $\mu$m.

The rough-surfaced samples that were sandblasted and acid-etched without any coating showed Ra values of about 43.0 $\mu$m. With coatings obtained on these samples with the PLD process, they demonstrated an increase in the Ra values to 45.3 $\mu$m. The same sand-blasted, acid-etched samples with initial Ra values of 43.0 $\mu$m, upon coating done by plasma spraying, showed a decrease in the surface roughness to 35.5 $\mu$m.

### X-ray diffraction

XRD analysis helped to identify and characterize the coatings based on the presence of different CaP phases. The specimens coated by the PLD process had two phases—HA and TTCP. They were then intentionally prepared to be equally distributed to have 12 specimens that were predominantly HA, and 12 as a mix of HA and TTCP. The ratio of the mixture, the process of incorporating the TTCP phase, and the methodology are beyond the scope of this paper and hence are not discussed. The plasma-sprayed specimens (n = 12) were identified as predominantly HA without significant presence of other phases of CaP.

**Table 2** Level of bond strength (MPa)

| Specimen | n | Mean bond strength | Standard deviation |
|---|---|---|---|
| PLD–HA (rough) | 12 | 68.3 | 17.8 |
| PLD–HA/TTCP (smooth) | 12 | 55.2 | 21.1 |
| Plasma-sprayed (rough) | 12 | 17.0 | 2.8 |

Comparisons were statistically significant at the 0.05 level.

### Scanning electron microscopy

The SEM analysis of the smooth-surfaced alloy showed a smooth surface with minimal irregularities. On the other hand, the blasted, acid-etched alloy showed evidence of a very coarse, rough surface on the alloy. The PLD coatings were approximately 1- to 3-$\mu$m thick, and the plasma-sprayed coatings were about 30- to 50-$\mu$m thick.

Analyzing the coatings at 1000× magnification showed that the surface of the PLD coatings was very uniform in nature, with the crystals being homogenous. A few pores could be observed and seemed to be scattered along the surface of the coating. A layered pattern was not present, and the surface rather seemed like a lamination over the smoothly polished titanium alloy surface. For the rough-surfaced alloy, the coating seemed to be more homogenous and followed the contours of the rough surface. Some particles were quite evident and more prominent, and the surface morphology was very uniform (Fig 3).

The plasma-sprayed samples that had been coated only on the rough-surfaced alloy had a very uneven surface. The surface showed the presence of splats caused by the intensity of the particles hitting the substrate and rapid cooling thereafter. The surface had particles of uneven shapes and sizes and lots of what appeared to have been molten matter in between them. The shapes of the particles were not clearly identifiable, and the picture showed evidence of particles being present in layers of one over the other (Fig 4).

### Z-axis bond strength test

The bond strength test showed that the PLD coatings had significantly higher bond strengths than the plasma-sprayed coatings as displayed in Table 2 and illustrated in a graphical representation (Fig 5). The average bond strength of the PLD coatings (rough and smooth) was in the range of 55.2 to 68.3 MPa. The PLD samples with higher crystalline HA (HA without TTCP) showed the highest average bond strength of 68.3 MPa followed by the PLD samples with lower crystalline HA (HA with

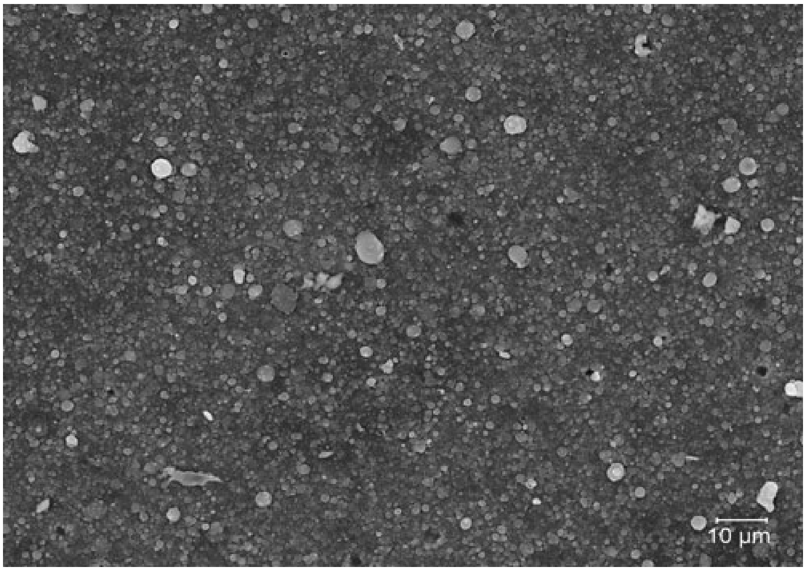

**Figure 3** SEM picture of an as-deposited pulsed laser deposited coating at 1000×.

TTCP) at 55.2 MPa. The plasma-sprayed coatings had an average bond strength of 17.0 MPa. SEM analysis of the bond strength-tested samples showed a very high frequency of multimodal fractures (fractures occurring at different layers) of the PLD coatings (Fig 6). As for the plasma-sprayed coatings, it was a very common trend to see complete delamination (single layer) of the coatings (Fig 7).

### Energy dispersive spectroscopy

The SEM energy dispersive spectroscopy was used to analyze the presence and absence of residual elements on the substrate following the fracture after bond strength testing. The PLD samples showed the presence of Ca and P, along with carbon (likely from the epoxy of the aluminum pull stud) suggesting a multimodal fracture. The plasma-sprayed samples did not show the presence of calcium or phosphorus elements along the tested area, suggesting a complete delamination.

## Discussion

Our study compared the HA coatings obtained by PLD with plasma spraying as to the alteration in surface roughness of the substrate alloy and the adhesive bond strength of the HA coatings to the alloy (Ti-6Al-4V). PLD HA coatings showed higher adhesive bond strength values compared with plasma-sprayed HA. The ability of the PLD method to produce thinner coatings

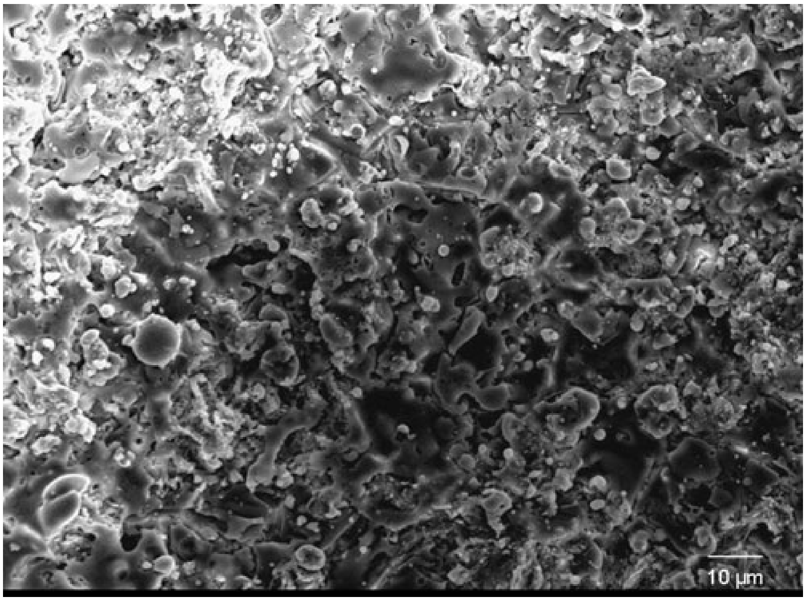

**Figure 4** SEM picture of an as-deposited plasma-sprayed coating at 1000×.

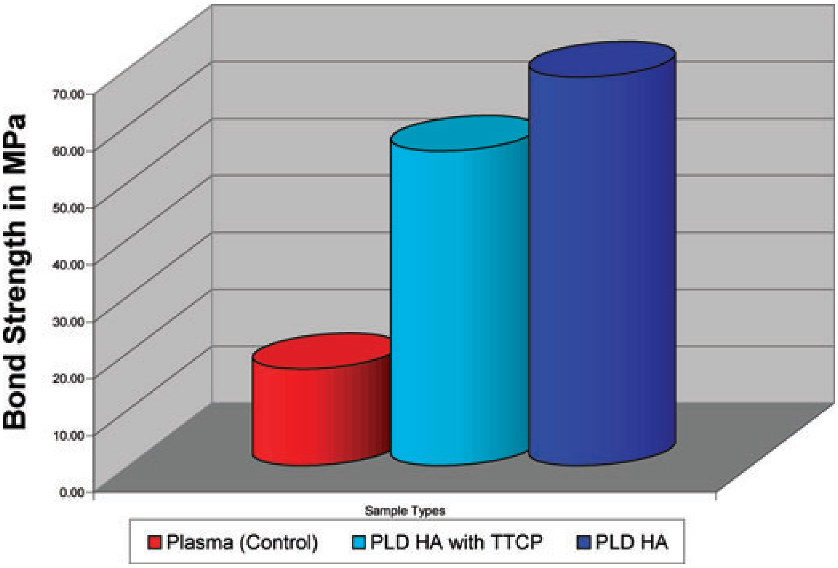

**Figure 5** Graphical representation of the average bond strength values of the different PLD coatings with the average bond strength of plasma-sprayed coatings. Plasma = plasma-sprayed hydroxyapatite coatings (rough-surfaced); PLD HA with TTCP = pulsed laser-deposited hydroxyapatite with tetra-calcium phosphate (smooth-surfaced); PLD HA = pulsed laser-deposited hydroxyapatite (rough surfaced).

may possibly contribute to a reduction in residual stresses along the coating–substrate interface. The accumulation of residual stresses in any coating has been shown to weaken its structure, making it more vulnerable when forces are applied. The PLD coatings in this study were less than 3 $\mu$m thick, whereas the plasma-sprayed coatings were approximately 50 $\mu$m thick. The increased thickness of a coating leads to an increased chance of the coating accumulating residual stresses. It has been established that the plasma-sprayed coatings develop residual stresses near the interface of the coating and the alloy. This is believed to be due to the mismatch in the thermal expansion coefficient that occurs when the plasma-sprayed HA coating rapidly cools from being in a molten mass of temperatures greater than 10,000°C to reach room temperature after the spraying process. The coefficient of thermal expansion of the metal is generally higher than the ceramic coating, and hence residual stresses develop along the interface during cooling.[14] Localized accumulations of stresses can promote a tendency for the coating to debond. Plasma-sprayed coatings have been reported in the literature to have very high cohesive bond strengths and rarely, if ever, fracture within the layers of the coatings; however, the adhesive bonding between the substrate and the coating is fairly low, increasing the chance for fracture at the coating–substrate interface during load-bearing situations.[15] The PLD process was performed at temperatures less than 800°C, which should reduce the effects of thermal expansion mismatch.

The coatings produced by the PLD process were uniform in nature at the surface level, with no significant difference in the amount of porosity or other surface irregularities. The multimodal fracture was a consistent pattern seen in both the rough- and smooth-surfaced PLD HA specimens after the bond strength tests. SEM analysis of these coatings showed that

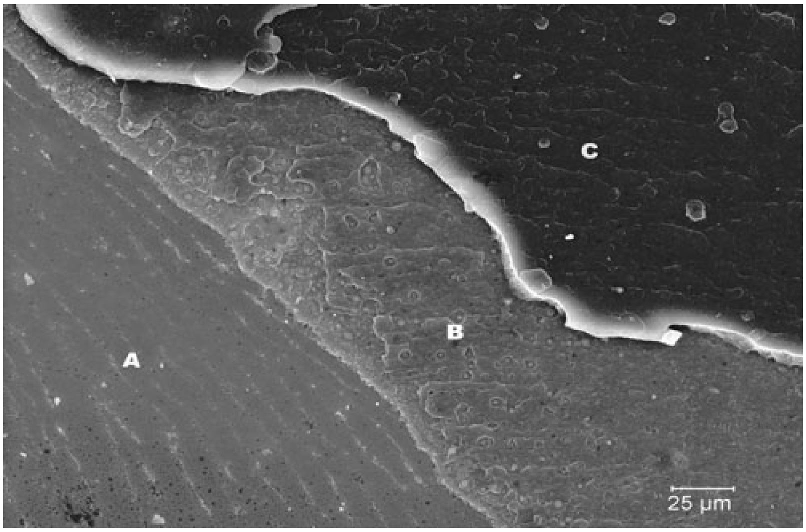

**Figure 6** SEM analysis of fracture area of a pulsed laser-deposited, hydroxyapatite-coated specimen at 500× magnification: (A) substrate, (B) HA coating, and (C) epoxy resin.

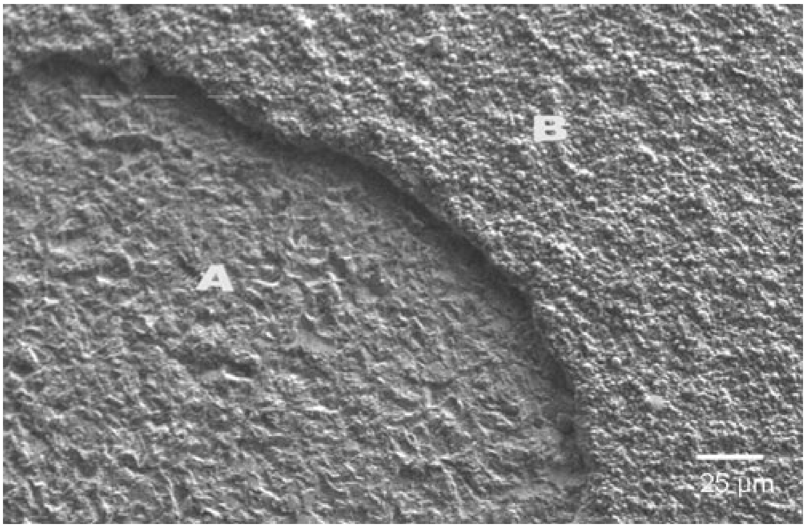

**Figure 7** Plasma-sprayed coating showing complete delamination of the hydroxyapatite coating from the titanium alloy substrate: (A) acid-etched, grit-blasted surface; (B) plasma-sprayed HA coating.

the fractures occurred along several modes, beginning with the interface of the coating and the alloy, spreading over to the interface of the coating and the epoxy resin and reaching the interface of the epoxy resin and the aluminum pull stud and hence was characteristically called a multi-modal fracture.[16]

As a part of the fracture analysis, EDS examination of the region was performed to determine the presence of any calcium or phosphate phases to assess if any cohesive failure (fracture within the layers of the coating) occurred within the PLD or plasma-sprayed coatings. The plasma-sprayed coatings showed complete delamination of the coatings from the alloy, whereas the PLD coatings fractured at multiple levels, indicating high adhesive strength of the coatings. The rationale for testing a rough- and a smooth-surfaced alloy without coating was primarily to assess the strength of the epoxy resin with the aluminum pull stud and to make sure that the epoxy resin did not fail under 80 MPa as recommended by the manufacturer.

The effect of surface roughness of the alloy and its role in successful osseointegration has been established. The rough-surfaced alloy used as substrate for coatings increases the adhesive bond strengths by achieving mechanical interlocking and retention of the coating. A rough surface also increases the surface area for greater volume of coating and for increased contact to withstand greater force transfer, thereby increasing the possibility of primary stability of the implant. It is not unusual for rough-surfaced titanium alloys to exhibit greater bond strength with their coatings, but in our case, an interesting finding was that the smooth-surfaced alloy with PLD HA coatings showed comparable bond strengths within their group. A reason for this could be the possible interaction between the phosphorus in the coating and the titanium in the alloy in forming a titanium phosphide compound. This establishes a chemical bond between the coating and the alloy, and this typically is not the case with plasma-sprayed HA coatings, as they mostly rely upon the mechanical bond between the coating and the rough-surfaced alloy.[17] Though this aspect of the material was not analyzed, this might be a possible explanation for the intimate contact observed between the substrate and the coating in the case of the PLD coatings.

Although our study used flat titanium alloy disks for this novel method of applying an HA coating, it is possible to coat complex structures like screws and cylinders. Within the vacuum chamber lies the substrate mount, which moves up and down, because the coating was desired only on one surface. It is possible for the mount to rotate and revolve within its axis, thereby rendering this method feasible for deposition of coatings on complex structures. PLD of HA to coat titanium alloys is an investigational method at this time and its applicability in commercial implant coatings has not been evaluated.

## Conclusion

HA coatings obtained by the PLD method show greater adhesive bond strength compared to the plasma spraying method of obtaining coatings. The pulsed laser-deposited coatings do not alter the surface roughness of the substrate alloy significantly, which is an advantage for increasing the potential for bony ingrowth and thereby having a greater bone-to-implant contact and rapid osseointegration. The thin-pulsed laser-deposited coatings have greater adhesive bond strengths to the substrate and tend not to delaminate upon testing like the plasma-sprayed coatings. These properties of pulsed laser-deposited coatings may increase their stability in load-bearing situations and allow the coating to withstand greater placement torques without causing much damage to the coatings.

## Acknowledgment

This research was supported by NIDCR grant # ROI DE 013952-04.

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