Determination of Adhesion Strength of Discrete Nanocrystalline HA Deposition Using Atomic Force Microscopy

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Introduction: Hydroxyapatite [HA: $Ca_{10}(PO_4)_6(OH)_2$] is a biocompatible material with chemistry similar to the mineral content of bone. HA coatings are commonly used on metallic orthopedic implants to enhance its surface osteoconductivity. BoneMaster™ and Discrete Crystal Deposition (DCD) are proprietary solution-based technologies of Biomet Inc. that can be used to obtain nanocrystalline HA deposition on metallic substrates. Tests recommended by ASTM standards (ASTM 1147-05 and 1044-05) to determine the adhesion strength of calcium phosphate coatings to metallic substrates may not be effective for thin coatings or discrete crystal depositions like those obtained with BoneMaster™ and DCD processes. In the present study, an alternative method has been proposed for using atomic force spectroscopy to characterize the adhesion strength of discrete nanocrystalline HA depositions on metallic substrates.

Materials and Methods: BoneMaster™ or DCD processes were used to obtain a discrete nanocrsytalline HA deposition on Ti-6Al-4V discs. Ten discs (25 mm dia, 6.4 mm thick) were coated with BoneMaster HA process and three discs (20 mm dia, 1.5 mm thick) with DCD process. Atomic force microscopy (AFM) was used to determine the adhesion strength of HA crystals to the metallic substrate. AFM (PicoForce, Digital Instruments) with a diamond-coated SiN cantilever was used to dislodge individual crystals of HA depositions obtained with BoneMaster™ or DCD processes. The individual HA crystals were located and variable forces were applied until the particle was dislodged from the surface, and the corresponding force value was recorded. In addition, the region was imaged to analyze the substrate surface area that the crystal occupied before it was dislodged. The shear stress was subsequently calculated as the ratio of the force required to displace the crystal and the surface area that the crystal occupied. Total of fifty (50) and thirty (30) data points were analyzed for BoneMaster™ and DCD processes respectively.

Results: The average shear adhesion strength between the HA crystals and metallic substrate for deposition obtained with BoneMaster™ process was 1.28 +/- 0.12 GPa, while that using DCD process was 1.52 +/- 0.29 GPa. Figure 1 shows the discrete nanocrystalline HA deposition obtained with BoneMaster™ process. Figure 2 shows a HA crystal from this deposition that was displaced using force spectroscopy. The surface area of the original crystal location was subsequently used for estimating the shear adhesion strength of the HA particle to the metallic substrate.

Conclusions: Conventional methods for analyzing the adhesion strength of coatings as described in ASTM standards may not be applicable to thin coatings and discrete nanocrystalline HA depositions on metallic substrates. This study demonstrated that atomic force spectroscopy is an alternative method for analyzing the adhesion strength of such depositions. Moreover, the results showed that the shear adhesion strength of nanocrystalline HA depositions on metallic substrates obtained with BoneMaster™ or DCD processes were substantially higher than that accepted for plasma-spray-HA coatings on metallic substrates (20 MPa).

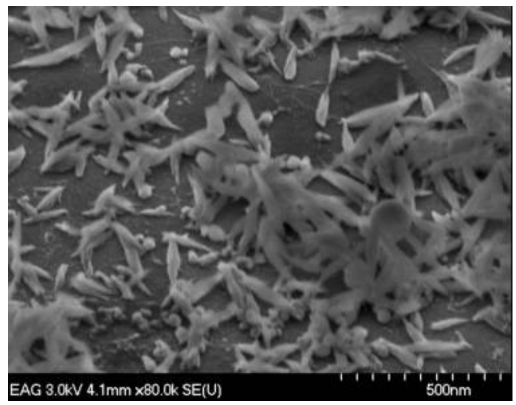

Figure 1. SEM image of discrete nanocrystalline HA deposition obtained using BoneMaster™ process.

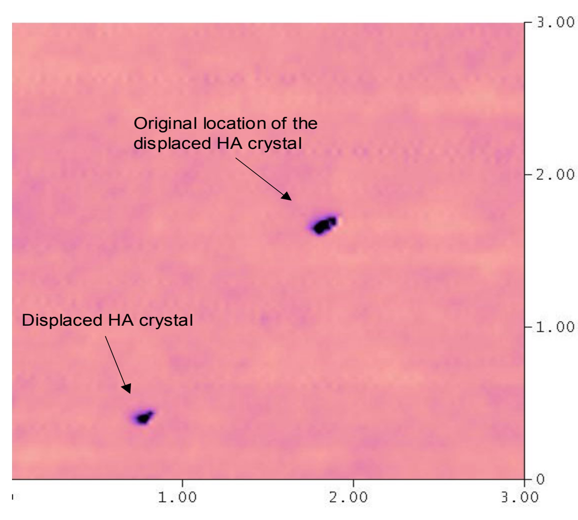

Figure 2. High-resolution de-convoluted image showing a HA crystal displaced using atomic force spectroscopy.