Significant Reduction of the Mechanical Strength of the Plasma Sprayed HA Coatings Soaked in Bovine Serum

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Introduction: Plasma sprayed hydroxyapatite (PSHA) coating is widely applied to metal implants due to its bone bonding capability. The commercially available PSHA coating may contain more than 95% HA, among which approximately 40% can be in an amorphous state (crystallinity of 60%). The amorphous HA is metastable and may dissolve preferentially after contact with the aqueous solution. This may result in the reduction of the adhesion and cohesion of the PSHA coating. Clinical follow-up studies (7-10 years) revealed a significant number of HA coated acetabular cups with mechanical loosening. This loosening is due to the coating resorption and delamination at the coating/metal interface. The as-received PSHA coating may show good adhesion at the time of implantation, but may not retain adhesion after implantation *in vivo* for a prolonged time. This poses the question of whether a permanent HA coating is necessary and sufficient (strong enough) for the insurance of the implant longevity once short-term bone bonding is achieved. The purpose of the current study is to evaluate how the dissolution of the unstable component (mainly amorphous phase) may affect the mechanical performance (tensile, shear, fatigue and abrasion properties) of the PSHA coating. The *in vitro* test may provide valuable insight into the performance of PSHA coating *in vivo*.

Methods: Ti6Al4V disks were coated on one side with plasma sprayed HA. All disks were grit-blasted prior to the HA coating process. Tensile (1" (ϕ) x 0.5" (H)), shear (0.75" (ϕ) x 1" (H)) and abrasion disks (1.5" (ϕ) x 0.5" (H)) were prepared for mechanical tests. PSHA coated tensile (Te) and shear (Sh) disks (N=10 for each group) were tested in accordance with ASTMF1147-99 and ASTMF1044-99. Shear fatigue of the PSHA coating was tested according to ASTM1659-95. Abrasion disks were tested by employing a pin-on-disk tester with a vertical load of 200N (UHMW polyethylene pin is 0.7"(L) x 0.375" (ϕ). In addition, PSHA coatings (N=5 per group) were soaked in supplemented α-calf bovine serum, containing 1.5mM Ca^{2+}, 0.5mM PO_4^{3+} and 1.5mM Mg^{2+}. The pH of the serum was adjusted to slightly acidic (pH=6.5). The test disks were soaked in 50ml of supplemented bovine serum for 48 hours at 37 °C with the serum being refreshed every 12 hours. Phase content and morphology of the PSHA coatings were examined by x-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). Ionic release (Ca and P) was monitored by atomic absorption (AA) and photospectrometer.

Results / Discussion: Figure 1 shows the XRD pattern of plasma-sprayed HA coatings before (AS-R) and after soaking in the serum (soaked). Dissolution of the amorphous component is marked by the disappearance of the hump between 27° and 35°(2θ). Figure 2 shows the net releases of the Ca^{2+} and PO_4^{3+} while soaking in the serum. The pH of the solution also increased (>6.5) during the immersion time. SEM (Figure 3) shows the soaked PSHA coating became more porous on the surface. Tensile and shear tests (Figure 4) show that the mean tensile/shear strength of the PSHA coatings suffered a more than 50% reduction after the 48h soaking. The shear fatigue test also demonstrated dramatic reduction of the fatigue life of the soaked PSHA coatings. The soaked PSHA coating became more fragile and generated more particles in comparison to the as-received PSHA coatings when subjected to abrasion testing.

Adhesion of the PSHA coatings was mainly dependent on the mechanical locking at the coating/metal interface. Most of the amorphous phase exists at the coating/metal interface due to the rapid metal substrate cooling during plasma spraying process. Release of the amorphous phase severely impaired the mechanical strength at the coating/metal interface, causing a reduced tensile/shear strength as well as lower fatigue life. On the other hand, the cohesion of the PSHA coating is largely dependent on the integrity of the amorphous matrix, in which the unmelted HA particles reside. Dissolution of the amorphous matrix weakened the binding of the adjacent HA particles.

Caution should be taken when extrapolating the current data to the *in vivo* condition. Even though the pH (6.5) of the supplemented bovine serum is only slightly lower than physiological condition (pH7.2-4), the dissolution of the amorphous phase may take longer to be equivalent to the *in vitro* test. Once bone bonding occurs directly onto the coating *in vivo*, the dissolution rate of the amorphous phase may be reduced. Regardless of the different dissolution rates of the amorphous phase *in vivo* and *in vitro*, the adverse effect of the dissolution of the amorphous phase on the mechanical stability of the PSHA coating is clearly identified in the current study.

Conclusions: Dissolution of the amorphous component in PSHA coating significantly reduced the coating adhesion and makes the coating prone to generate more particles.

Fig. 1

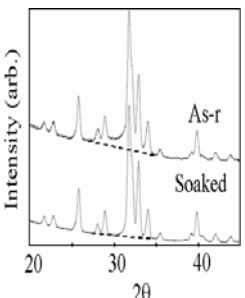

Fig. 2

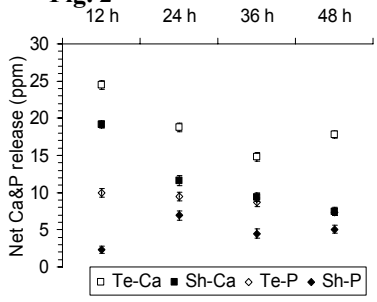

Fig. 3

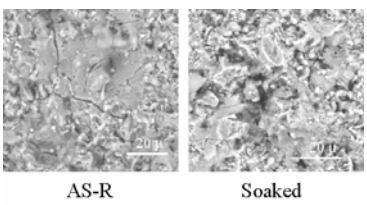

Fig. 4