# Torsional fatigue resistance of plasma sprayed HA coating on Ti–6Al–4V

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The torsional strength of plasma sprayed hydroxyapatite (HA) coatings was studied under static and cyclic loading. The torsional shear tests were conducted in a frustum test device developed in this laboratory, which adapted to various coating thicknesses. The interfacial fatigue resistance was measured in terms of interfacial fatigue strength defined as the average maximum stress ($\tau_{fmax}$). A staircase fatigue method was employed to determine the interfacial fatigue strength; this method resolved the uncertainty in detecting coating failure during torsion fatigue. The values for coating shear strength and shear fatigue strength obtained from the torsional tests did not differ from those obtained by previous tensional shear tests in this laboratory. The fatigue strength of one million cycles was about 35% lower than static shear strength. This finding might be used for estimating fatigue life span without cyclic loading tests.


## Introduction

Calcium phosphate coatings are popular in dental and orthopedic metal implants because they achieve good osteointegration [1–4]. The strength of a hydroxyapatite (HA)-coated implant and its long-term stability in the human body much depends on the bonding strength between the coating and the metal substrate. In fact, it has been found in clinical applications that mechanical failure usually occurs at the HA coating/substrate interface [5, 6]. It is desirable that the interfacial strength is sufficiently high to sustain complex loading without coating spallation. Mechanical evaluation of the coating/metal interface has to be performed before coated implants can be reliably used in clinical applications.

Commonly, static strength is used to represent coating integrity; however, orthopedic or dental implants in a living body are sustained cyclic loading. Fatigue resistance of coatings is especially important for the implants in younger and more active patients. Although fatigue behavior of HA-coated implants has attracted much attention in recent years, most have addressed fatigue strength of coated substrate, not coating bond strength [7–12]. Study of HA coating interfacial fatigue strength is quite limited.

A simple and advantageous method for testing interfacial shear strength of coatings has been developed in this laboratory. Called the frustum shear test method, this approach ingeniously avoids misalignment, and easily handles samples with various coating thicknesses [13]. This method has been successfully used to evaluate coating fatigue shear strength under tensional loading [13]. Note that studies indicate torsion is the common load on stem of hip joint implants *in vivo*; such torsion is generated in daily activities such as getting up from a chair or stair climbing, and sometimes the torque can reach 40 Nm [14, 15]. To simulate the loading on stem of hip joint implants, torsional cyclic loading was applied in the testing. Torsion shear tests can generate pure shear, eliminating normal stress that is commonly associated with other types of shear testing.

## Experimental procedure

### Materials

Each cylindrical Ti–6Al–4V specimen with a radius of 6 mm was machined with a taper angle of 3° for each 6 mm in length. The surface of Ti alloy specimens were sandblasted by $Al_2O_3$ powder and then ultrasonically cleaned in ethanol before being coated. HA starting powder with an average diameter of 50–70 μm was plasma sprayed onto the constrained taper parts of Ti alloy specimens. The plasma gas was $N_2$, and the arc current and voltage ranges were 370–420 A and 60–80 V, respectively. 90 and 200 μm of coating thickness were obtained by plasma spray. For studying the effects of crystallinity, some specimens were heat treated at 650 °C for 2 h. All the coated specimens were ultrasonically cleaned in deionized water before binding with inner

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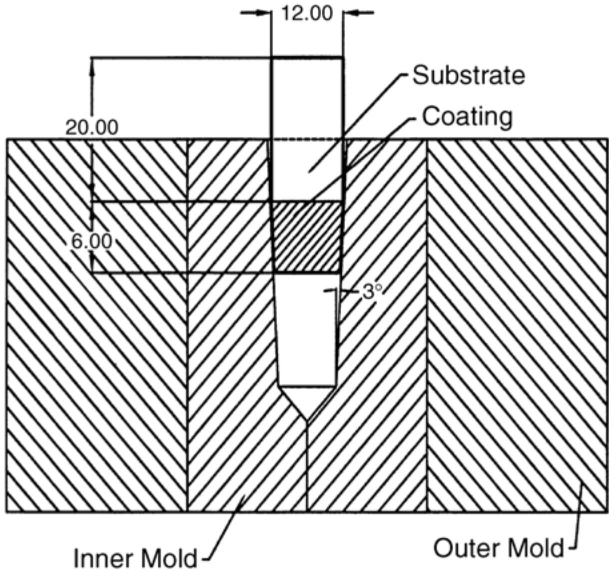


*Figure 1* Schematic illustration of the frustum test set-up for shear test.

mold with adhesive film. FM 1000 adhesive film (Cytec Fiberite Inc., USA) with a thickness of 0.01 inch was used to bond the coating onto the surface of the inner mold. The optimal curing conditions were 162 °C for 2 h. All mechanical tests were performed on a MTS 858 servo-hydraulic machine at room temperature in air.

## Testing

The frustum testing device is schematically illustrated in Fig. 1, a detailed description can be found in Zhang *et al.* [13]. Both static and cyclic tests were conducted in order to evaluate their correlation. The static torsion tests were conducted under angle-controlled mode at a rate of 2°/min to obtain the maximum torque required for damaging the coating/substrate interface. Also, static tension tests were performed with a displacement-controled mode at a rate of 0.3 mm/min in order to compare with previous testing data reported in Zhang *et al.* [13]. Each strength value is the average of five specimens. The tensional shear strength ($\tau_{ten}$) and torsional shear strength ($\tau_{tor}$) were calculated according to the following formulas.

$$\tau_{ten} = \frac{F}{A} \tag{1}$$

where $F$ is the maximum force applied in the test, and $A$ is the coating area, 230 mm$^2$.

$$\tau_{tor} = \frac{T}{RA} \tag{2}$$

where $T$ is the maximum torque, $A$ is the coating area, 230 mm$^2$, and $R$ is the radius of the cylindrical sample. The difference of radius was negligible because the maximum radius difference in the taper region was $\sim 5\%$.

The fatigue tests were run sinusoidally in torque-controled mode with fixed torque amplitude ($T_a$) of 15 Nm with a frequency of 10 Hz for one million cycles. The fatigue resistance was examined by disassembling the testing fixture because of difficulty in determining torsion failure during cyclic loading. After the prescribed number of cycles, the inner mold with specimen was heated to 450 °C for 1 h. The adhesive between specimen and inner mold was burnt out during heating; thus specimen could be easily removed and it could be determined whether the coating had failure. The fatigue strength was determined by the staircase method [16, 17]. Change of maximum shear stress in staircase method was realized by changing the mean torque ($T_m$) with a step size of 2.5 Nm, i.e. 1.8 MPa in maximum stress $\tau_{max}$. In this work, the fatigue strength was defined as statistical average maximum stress $\tau_{max}$, and was indicated as $\tau_{fmax}$. Each test case contained 24 specimens. The *t*-test was used to determine significant differences in average fatigue strength among the four samples. Significance was set at $p < 0.01$.

Semi-quantitative analyses of the chemical composition of the coating–substrate interface were also conducted by energy dispersive X-ray spectroscopy (EDS) in a scanning electron microscope (SEM), JEOL 6300. Data acquisition was conducted at an accelerating voltage of 15 kV for 300 s on the carbon-coated samples used for the morphology analysis.

## Experimental results

Table I compares shear strength obtained by the static torsion tests and the tension tests. The results reveal that the interfacial shear strengths obtained by torsion tests ($\tau_{tor}$) and by tension ($\tau_{ten}$) are nearly identical. This comparison indicates that the shear strength of interface is rather isotropic. Table I shows that the samples with thick coating (200 μm) exhibit lower shear strength than those with thin coating (90 μm) by around 20%. Results also indicate that the post-spray heat treatment did not obviously affect bonding strength. After the treatment, the bond strength decreased by 4% for 90 μm coating, while there was no evident effect for the 200 μm coating.

Fig. 2 shows the typical torsion angle change as a function of loading cycles. A sudden change of curve slope is expected when adhesion fails between the HA coating and the Ti substrate. The recorded experimental curve (Fig. 2), however, does not indicate the cycle numbers at which coating failure occurred. Thus, all the data of fatigue relied on examining the coating integrity after $10^6$ cycles. The data of fatigue tests are exhibited in ''staircase'' fashion (Fig. 3). The O and X represent non-failure and failure cases, respectively. The average fatigue strengths at $10^6$ cycles ($\tau_{fmax}$) for these samples are shown in Table II, was and were calculated by the following method [16]:

$$m = S_0 + d\left(\frac{A}{n} \pm \frac{1}{2}\right) \tag{3}$$

TABLE I Static interfacial shear strength by torsion ($\tau_{tor}$) and by tension ($\tau_{ten}$) ($\pm$ S.D.) in MPa

| | $\tau_{tor}$ | $\tau_{ten}$ |
|---|---|---|
| 200 μm, as-sprayed | 30.9 ± 2.62 | 30.2 ± 1.80 |
| 200 μm, as-treated | 31.2 ± 2.13 | 28.3 ± 1.67 |
| 90 μm, as-sprayed | 40.5 ± 2.58 | 39.5 ± 1.68 |
| 90 μm, as-treated | 38.7 ± 1.60 | 38.7 ± 2.10 |

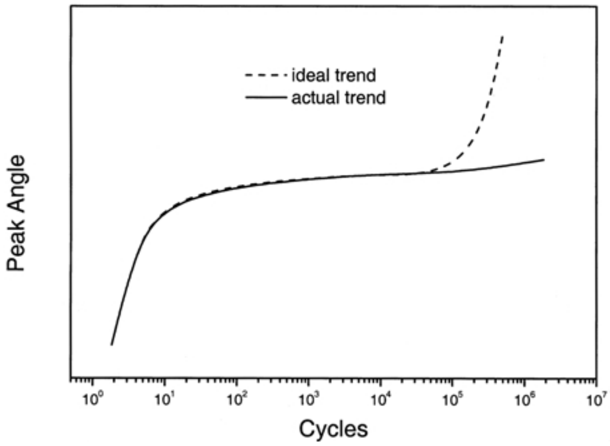


*Figure 2* Torsional angle change with the cycle number during the fatigue tests. The real curves does not indicate the cycle number when fatigue failure occurs.

where $m$ is the average fatigue strength (specifically, $\tau_{fmax}$). $S_0$ is the lowest stress used in the test and $d$ is the stress increment or step size. $n = \Sigma_{i=0}^{z} n_i$ is the total of less frequent events, where $n_i$ is the number of the less frequent events at $i$-th stress above $S_0$; $i$ is the coded stress level ($i = 0$ for $S_0$); $z$ is the number of stress levels above $S_0$. $A = \Sigma_{i=0}^{Z} i n_i$. In Equation 3, $+1/2$ is used when the less frequent event is a runout, otherwise $-1/2$ is used.

The standard deviation was calculated by the following equations.

$$\text{S.D.} = 1.62\, d \left\{ \frac{B \sum_{i=0}^{Z} n_i - A^2}{n^2} + 0.029 \right\},$$

$$\text{when } \frac{B \sum_{i=0}^{Z} n_i - A^2}{n^2} \geq 0.3 \qquad (4)$$

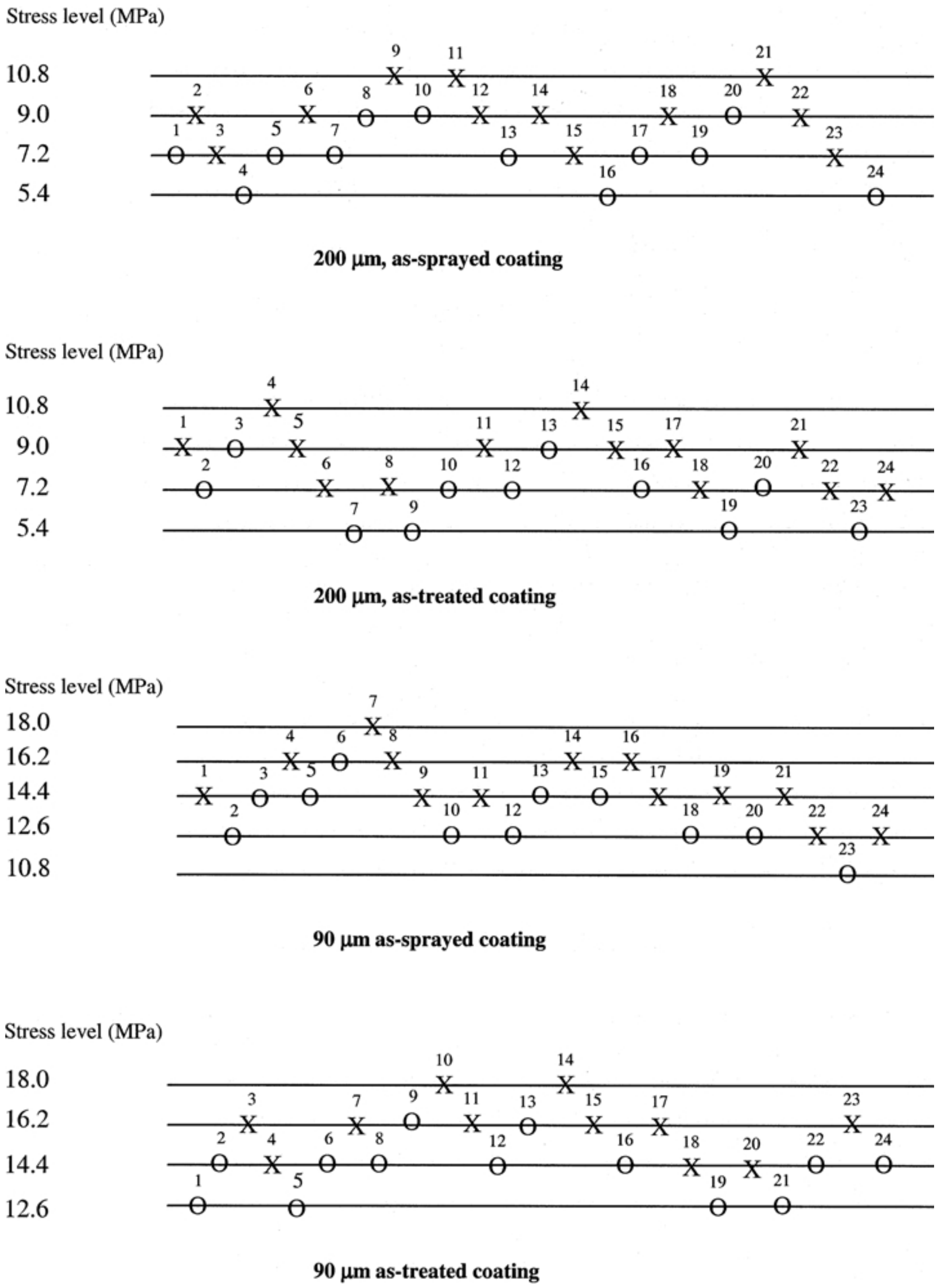


*Figure 3* Fatigue test results in ''staircase''. The stress level shown is the mean shear stress converted from torque value in individual fatigue test. The step size is 1.8 MPa. ''X'' indicates broken test pieces at $10^6$ cycles; ''O'' indicates unbroken test pieces at $10^6$ cycles.

TABLE II Average fatigue strength of tested samples ($\tau_{fmax} \pm$S.D.) in MPa

| | $\tau_{fmax}$ |
|---|---|
| 200 μm, as-sprayed | 18.9 ± 1.54 |
| 200 μm, as-treated | 18.6 ± 1.58 |
| 90 μm, as-sprayed | 24.4 ± 1.87 |
| 90 μm, as-treated | 23.8 ± 1.60 |

or

$$\text{S.D.} = 0.53\,d, \quad \text{when}\ \frac{B\sum_{i=0}^{Z} n_i - A^2}{n^2} < 0.3 \qquad (5)$$

The results revealed that the fatigue strength of the 90 μm coating is significantly higher than that of the 200 μm coating with $p$ value less than 0.001. For the same coating thickness, however, the as-sprayed sample and the as-treated sample exhibit significant differences in average fatigue strength with $p$ value of 0.06–0.07.

The exposed fresh substrate surfaces resulting from coating spallation were examined by SEM and EDS. The typical micrograph of the fractured surface is shown in Fig. 4(a). The shear failure separated the HA coating and titanium substrate while the surface was partially covered with a thin HA coating. EDS mapping of Ca and P identified the remained HA coating even after the coating had been spalled off macroscopically, and the Ti signal was detected from the complementary area in the map (Figs. 4(c)–(e)). Fig. 4(b) reveals very weak carbon signals evenly distributed over the whole examined area. There is no evidence of FM 1000 adhesive penetration to the interface and the test results were not affected by the adhesive penetration.

## Discussion

Table III shows the comparison of all shear testing data of HA coatings obtained in this laboratory, including the

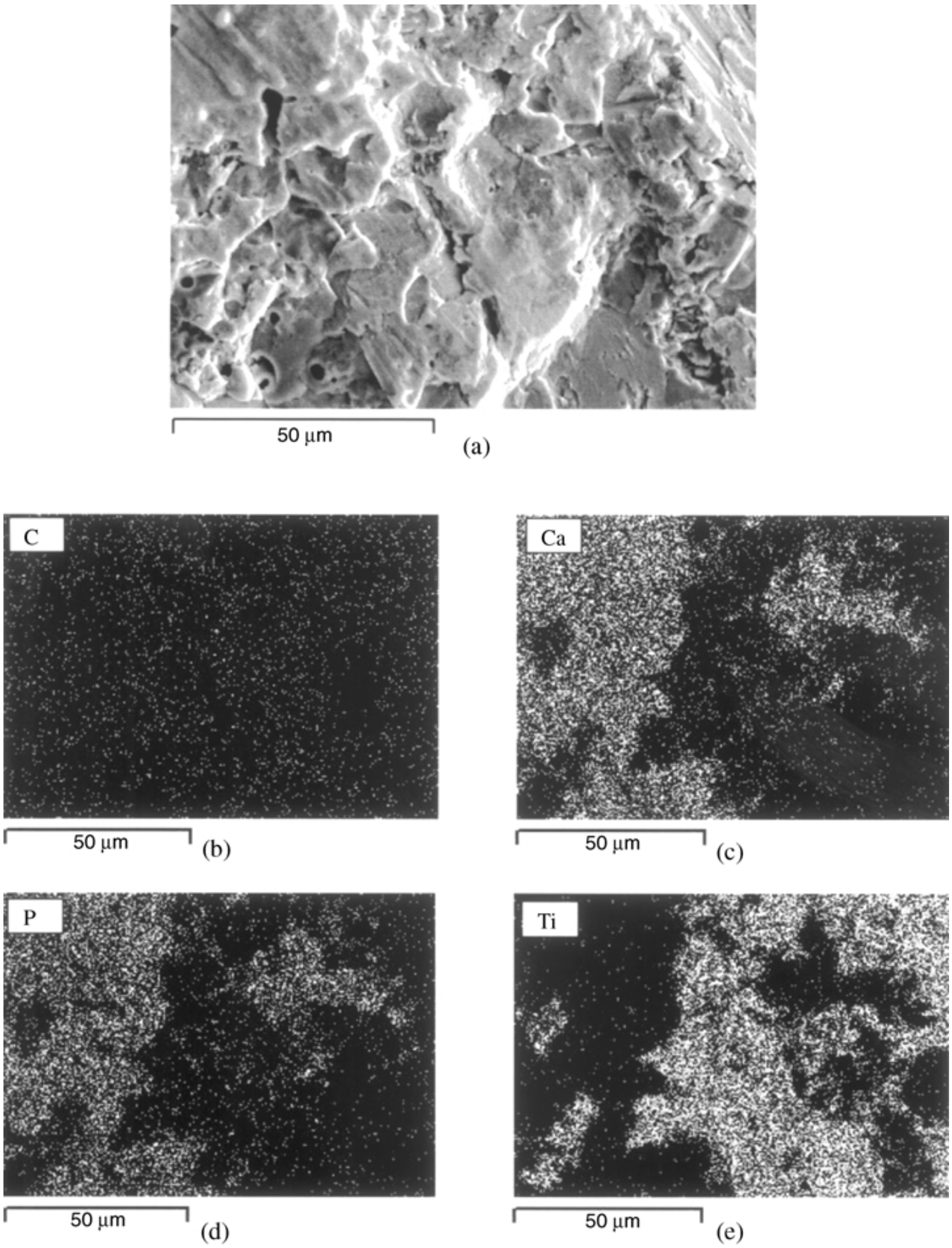


*Figure 4* Typical SEM micrographs and EDX results of the fractured interface.

TABLE III Summary of static shear strength and fatigue data in MPa

| Coating thickness | Static shear strength ($\tau_0$) | | | Fatigue data | | $\tau_{fmax}/\tau_0$ |
|---|---|---|---|---|---|---|
| | By tension ($\tau_{ten}$) | By torsion ($\tau_{tor}$) | By tension (ref. [13]) | By torsion ($\tau_{fmax}$) | By tension (ref. [13]) | |
| 90 µm | ~ 39 | ~ 39 | ~ 34 | ~ 25 | ~ 25 | 0.63 |
| 200 µm | ~ 29 | ~ 31 | ~ 28 | ~ 19 | ~ 20 | 0.67 |

previous work by Zhang *et al*. [13]. There is no obvious difference in tensional and torsional tests. This indicates that the normal stress level induced in the frustum tensional shear test does not affect the shear test results. Since it is more readily controled during testing, tensional shear tests might replace the torsional tests.

The data in Table III shows an interesting correlation between static and fatigue strength, which is represented by the ratio of $\tau_{fmax}/\tau_0$. The torsional fatigue strength is around 65% of static strength due to accumulative damage regardless of coating thickness. After analyzing the previous tensional shear fatigue data [13], we found that the $\tau_{fmax}/\tau_0$ value is also around 60–75%, consistent with the results in this study. It indicates that the cyclic loading (either tension or torsion) can result in the same extent of fatigue damage and that the damage can be estimated by the coating shear strength for static loading. This finding can be used in designing coated implants and in estimating the life span of implants for clinical applications.

Table III also indicates that coating thickness is a critical factor influencing coating shear and fatigue strength. It is clear that the 200 µm coatings have lower $\tau_0(\tau_{tor}$ or $\tau_{ten})$ and lower $\tau_{fmax}$ than those of the 90 µm coatings. Thin coatings have higher interfacial fatigue resistance than thick ones. This can be partially attributed to residual thermal stress in coatings induced by the plasma spray. Yang *et al*. found that the residual stresses (tensile) increased in proportion to the coating thickness [18]. In addition, the smaller amount of imperfection in thinner coatings may contribute to higher bond strength and fatigue strength.

The influence of heat treatment on the strength of plasma sprayed HA coatings, however, is quite controversial. Kweh *et al*. [19] concluded that heat treatment can improve in the mechanical properties of the coatings. This was reflected in the significant rise in micro-hardness and modulus of coatings and also the coating bond strength; in particular, heat treatment of 800 °C apparently showed these influences. Note that it is also possible that heat treatment introduces more cracks in the coatings, which enable epoxy adhesive to easily penetrate into the interface and thereby skew testing results. Tsui *et al*. [20] claimed that heat treatment at 700 °C for 1 h in air had a detrimental effect on adhesion strength of plasma sprayed HA coating [20]. They argued that phase transformation of amorphous to crystalline HA promoted by heat treatment was accompanied by a volume reduction in coating; thus a tensile stress was expected to arise in the as-treated coating. In the present work, the as-sprayed samples and the as-treated samples with same coating thickness do not show obvious differences in static and fatigue strength. This is consistent with that reported by Zhang *et al*. [13]. The damage theory due to volume change of phase transformation may not be applicable to plasma sprayed coatings, because existing pores and microcracks in plasma sprayed coatings could adapt to the volume change and release the subsequent internal stress.

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