Acoustic Emission Behavior on Fatigue Crack Characterization of Plasma Sprayed Hydroxyapatite Coated Layers

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ABSTRACT

In order to improve the adhesive strength of Hydroxyapatite (HAp) coating, the grit-blasting with Al_2O_3 powders and subsequently wet-blasting by HAp/Ti mixed powders were applied on Cp-Ti substrate. Two-layers of coating composed of HAp/Ti bond coat and HAp top coat were deposited by plasma spraying. HAp-coated specimen could survive up to 10^7 cycles, without spallation of HAp coating at the stress amplitude of 120 MPa under four point bending fatigue test. In order to clarify mechanical failure behavior of the coatings and Ti substrate, acoustic emission (AE) signals during an entire fatigue process were observed. Relationship between AE and crack growth behaviors of coated specimens was evaluated to predict the fatigue lives of coating layers. HAp top coat with HAp/Ti bond coat show high adhesive strength where most of damages occurred at the final stage of fatigue life.

1. INTRODUCTION

To improve bioactivity, Ti and its alloys often coated with Hydroxyapatite (HAp) have been widely used in orthopaedic and dental applications. In this case, the biocompatibility of implant materials is assured by HAp, while the mechanical properties are reinforced by the metal substrate. In previous work (1), grit blasting and wet blasting with HAp/Ti mixed powders and deposition of a HAp/Ti bond coat between HAp top coat and Ti substrate was developed. Application of mixed HAp/Ti bond coating could provide high strength of HAp top coat under tension-tension and compression-compression fatigue tests. In this study, the fatigue behavior of plasma sprayed HAp top coat with HAp/Ti bond coat layers on Cp-Ti substrate was investigated by four point bending condition. During the tests, AE behavior was monitored to discuss the damage process. Scanning electron microscope (SEM) observation and digital microscope were also carried out to reveal the morphologies of damage and to correlate the degree of damages in the coated specimen with the AE signals. Relationship between AE and crack growth behaviors of coated specimens was evaluated and used to predict the fatigue lives of HAp coated layers.

2. EXPERIMENTAL PROCEDURES

A rectangular shape of commercially pure titanium (Cp-Ti) grade 4 machined with size of $10 \times 50 \times 3 \text{ mm}^3$ is used as substrate materials and then were grit-blasted by Al_2O_3 powders with 700 μm in average particle size. The blasted substrates were then blasted again by de-agglomerated HAp/Ti mixed powders (3:2 volume ratios) of 100 ml fed into 400 ml distilled water with a wet blasting machine in order to generate a thin layer composed of HAp/Ti on the surfaces of substrates and to improve an appropriate surface roughness for HAp plasma spray coating. A plasma spray coating was conducted using a commercial plasma spray machine operated at 36 kVA (60V, 600A) with a spraying distance of 30 cm under shield flow of Argon gas. The two-layers

of coating composed of HAp top coat with HAp/Ti bond coat were deposited on wet blasted Cp-Ti specimen with an average coating thickness of 50 μm and 100 μm respectively. Fatigue tests under four point bending with 20 mm inner span and 40 mm outer span by using sinusoidal wave form with a stress ratio, $R=0.1$ and a frequency of 20 Hz were conducted in the controlled room at 23°C and 55% relative humidity. In order to clarify fatigue failure behavior and to predict the fatigue life of the coating layers, AE signals during the entire fatigue process were observed on both and single sides coating specimens. AE system (AE9922 combined with AS-712) with a gain value of 30dB and a threshold value of 0.65V was used to monitor the AE signal throughout the fatigue tests. SEM and digital microscope observation was also conducted to observe damage processes of the HAp coated specimens to identify the correlation between AE signal and damage morphology. The relationship between AE signal, average crack propagation length and number of fatigue cycles of coatings were used for the fatigue life prediction of HAp top coat and HAp/Ti bond coat specimen.

3. RESULTS AND DISCUSSIONS

From the cross-sectional observations of as-sprayed specimen, it is clearly seen that the coated layers were tightly attached to the Ti substrate and also to the bond coat layer. S-N curves obtained almost coincide for as-wet blast Cp-Ti and HAp coated specimens in Fig. 1. As-wet blasted and HAp coated specimens could survive up to 10^7 cycles without delamination and spallation of HAp coating at the stress amplitude of 120 MPa. The examples of AE signals, AE peak amplitude, during the fatigue test under a stress amplitude of 220 MPa are shown in Fig. 2. The AE signals under other stress amplitude conditions indicated similar tendency to Fig. 2. AE signals of as-wet blast specimens were mainly detected in the early and final stage and hardly detected in the intermediate stage of fatigue life. These signals may correspond to fatigue behaviors of Cp-Ti which may be cause from dislocation movements, the plastic deformation and the crack propagation of Cp-Ti.

Fig.1 S-N curves for as-wet blast Cp-Ti, HAp coated specimens, HAp top coat and HAp/Ti bond coat prediction

Fig.2 AE peak amplitude during the fatigue test under stress amplitude of 220 MPa for as-wet blast Cp-Ti (a) and HAp coated specimens (b)

Fig.3 The relation between fatigue behaviors of HAp coated specimen and AE signals during fatigue test subjected to tension loading

In HAp coated specimens, dense and high values of AE signals were mainly detected in the beginning and last stage of fatigue test and occasionally detected during the middle stage of fatigue life. AE signals generated from HAp coated specimens are denser and higher than those of as-wet blasted specimens, which may correspond to the formations of crack and the failure of coating layers during the beginning and last stages respectively while the AE signals during the intermediate stage may correspond to the propagation of crack in coating layers. As a former result (2), the fatigue life of HAp coated specimen under bending fatigue test is dominated by cracks from the tension side. Moreover, the delamination cracks in compressive side of coating obviously observed only at the final stage of fatigue tests and it is not affected their fatigue lives. In order to clarify the relation between AE signals and failure of coatings, a single side coating of HAp coated specimen was also conducted the fatigue test under stress amplitude of 220 MPa with AE monitoring in both tension and compression loading directions. AE signals from single side coating in tension fatigue loading indicated a similar tendency to both sides of coating as shown in Fig.2 (b) whereas AE signals from compression fatigue loading were detected in the early and final stages and scarcely detected in the intermediate stage of fatigue life.

In order to investigate fatigue life of HAp top coat and HAp/Ti bond coat, the relationship between AE signals and fatigue damage in coating layers subjected to tension loading was evaluated. In-situ observation of the specimen during the fatigue test with AE measurement was carried out. A digital microscope (VHX-1000) was placed in front of the edge of the specimen which was polished until 1 μ m of diamond paste. Average crack propagation length in coating layers was measured between the inner span of four point bending during each interrupted fatigue cycles. AE signals from interrupted fatigue test in Fig. 3, (a) reveals the similar tendency with the continuous test which was shown in Fig 2 (b). From the observation, it could be noticed that some cracks completely propagated through HAp top coat after 3,000 cycles and then most of cracks reached HAp/Ti bond coat layer after 9,000 cycles of interrupted test at the same time some

Fig 4 The relation between accumulated AE peak amplitude and accumulated average crack in HAp coating layers

cracks had already propagated into bond coat layer. Most of cracks reached Ti after 30,000 cycles thereafter cracks initiated and propagated into Ti substrate.

The relation between fatigue failure behaviors of HAp coated specimen and AE signal can be summarized by divided into three stages. During first and second stage, micro-cracks occurred in HAp top coat and HAp/Ti bond coat layers respectively and generated dense of AE signals as presented in Fig.3 (b). In third stage, crack initiated and propagated into Ti substrate starting from the edge corner of specimen without deflected to both interface layers which induced the macro-cracks in the coating layers in the final stage of its fatigue life. Therefore, The AE data referred to the damage crack in the coating layers during first and second stages were used to find the relation between crack propagation length and AE signal. The relation between accumulates AE signal and accumulated average crack length in HAp coating layers was plotted and fit in order to predict an average crack growth in coating layers as shown in Fig. 4. From the fit curves, it can be divided into two stages; first is the crack propagation in HAp top coat from 0~100 μ m and second is the crack propagation in HAp/Ti bond coat from ~100~150 μ m.

In order to predict fatigue life of coating layers, it is noted that the influent of AE signals from coating layers in compression side is possible to comprise. The values of AE signal accumulation after average crack propagated through HAp top coat and HAp/Ti bond coat calculated from the fit curves in Fig 4 were used to predict fatigue life of coating layers. The fatigue prediction in Fig. 1 presents very short fatigue life may due to the inclusion of AE signals from compression side of both side coating specimen which might be generated during the early stage of fatigue test.

4. CONCLUSION

Base on the in-situ observation and experimentally determined correlations between average crack propagation length in HAp top coat and HAp/Ti bond coat layers, AE signals and numbers of fatigue cycles, AE signal accumulation methods have been developed for estimating fatigue life of coating layers. HAp top coat and HAp/Ti bond coat presented very short fatigue life compared to the Ti substrate. The inclusion of AE signals from compression side of both side coating specimen which might be generated at the area of upper support pins may be caused of the shorten fatigue life prediction from accumulation of AE signals. However, HAp top coat with HAp/Ti bond coat show high adhesive strength where the delamination and the spallation of the coating layers was not observed until the final stage of fatigue life.

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