EFFECTS OF DYNAMICALLY ION MIXED THIN COATINGS ON FATIGUE DAMAGE PROCESSES IN TITANIUM ALLOYS

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Adherent amorphous NiTi and SiC coatings have been engineered by the Dynamic Ion Mixing technique in order to improve the fatigue resistance of Ti6Al4V and Ti6246 titanium alloys. Both treated substrata have been tested, in air, at room temperature in the Low Cycle Fatigue range. DIM NiTi and SiC films modify the surface deformation mechanisms of fatigued materials and suppress or delay microcrack initiation. Consequently significant fatigue life improvements have been obtained. These effects depend on the nature of the film and on the applied cyclic stress amplitude. They are discussed by considering the coating thickness and mechanical properties, and also the substrate deformation and damage mode.

INTRODUCTION

In titanium alloys as in most homogeneous metallic materials, the fatigue crack initiation occurs at the surface. In this work, a surface treatment method, the Dynamic Ion Mixing (DIM) technique, has been used in order to prevent crack initiation and to improve the fatigue resistance of titanium alloys. The DIM which involves a Physical Vapour Deposition process combined with a simultaneous high energetic ion implantation permits to obtain dense and homogeneous thin films in the order of the micrometer. The DIM leads to a gradual substrate – coating interface which ensures a very good adhesion. In this paper the effects of DIM NiTi and SiC coatings on the fatigue properties of both the Ti6Al4V and the Ti6246 titanium alloys will be presented. NiTi has been chosen for its superelasticity and SiC for its resistance to corrosion.

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EXPERIMENTAL DETAILS

DIM Coatings

DIM NiTi and SiC coatings, 0.2 to 1.5 µm thick were engineered by a sputtering method using a broad beam Ar^+ ions source of the Kaufman type. The growing films were simultaneously bombarded with 320 keV Ar^{++} ions which were produced, selected and accelerated in an implantor. A complete description of the apparatus was given previously (Jaulin et al.(1)).

Materials

The Ti6Al4V type titanium alloy (80 % of primary α phase) presents an heterogeneous globulized structure with large colonies of aligned coarse α platelets surrounded by small equiaxed grains. The Ti6246 alloy is a Ti 6Al 2Sn 4Zr 6Mo β-forged with a lamellar structure, the primary α platelets having lengths between 3 and 50 micrometers. Mechanical properties of both alloys are indicated in Table 1.

TABLE 1 – Mechanical properties of the two titanium alloys

	E (GPa)	A (%)	σ_{YS} (MPa)	σ_{UTS} (MPa)
Ti 6Al 4V	123	16	975	1035
Ti 6246	125	10	993	1061

Fatigue tests

Smooth cylindrical specimens with a diameter of 6 mm were used. They were polished up to a diamond paste of 0.25 µm before surface treatment. Fatigue tests were performed in air at room temperature in a symmetrical uniaxial push-pull mode ($R = -1$) under stress control ($\Delta\sigma/2 = \pm 750$ MPa or ± 850 MPa).

RESULTS

DIM NiTi and SiC coating properties

Previous Transmission Electron Microscopy and Secondary Ion Mass Spectrometry investigations have shown that DIM films are dense and homogeneous. Both coatings are characterized by an amorphous structure with some areas exhibiting a β-nanocrystallization for SiC films. Scratch tests have shown that both NiTi and SiC DIM films are adherent to titanium alloys. Micro-Vickers indentations have revealed the high ductility of NiTi coatings which are able to accommodate very high plastic deformations whereas the brittle nature of SiC films has been pointed out (Figure 1). Moreover, it has been shown that thicker is the SiC coating, greater is its brittleness (Peraud et al.(2)).To determine deformation and stress amplitudes reached in coatings during fatigue experiments, the knowledge of their Young's modulus is required. Measurements have been performed by a resonant frequency technique (Peraud et al.(3)) which leads to 105 GPa and 235 GPa (± 10 %) for respectively NiTi and SiC films. It must be noted that the value obtained for SiC is very low compared to the average value of 440 GPa obtained for crystalline SiC CVD coatings (Watkins et al.(4)).

Cyclic deformation and damage of untreated materials

At a stress amplitude of $\Delta\sigma/2 = \pm 750$ MPa, the Ti6Al4V is cyclically deformed in their elastic field and the number of cycles at failure is about 5,000 cycles. Moreover, regularly interrupted tests with SEM observations of specimen surface have shown that the crack initiation occurs early in the fatigue life, the number of cycles at failure being mainly determined by the crack propagation stage. At failure, the fatigue damage is characterized by a high number of microcracks, about 10 cracks per mm^2 (Demulsant et al.(5)).

For the Ti6246, the fatigue lives are 12,000 and 4,000 cycles at respectively $\Delta\sigma/2 = \pm 750$ MPa and ±850 MPa. The greater resistance of this second alloy is due to a better resistance to crack initiation. Indeed, it has been shown that the first cracks appear only at the half-fatigue life, and that the total cracks density at failure remains very small (0.8 crack per mm^2).

Moreover microcracks initiate within colonies either perpendicularly to the α grains or following the α/β interfaces for the Ti6Al4V alloy. For the Ti 6246 alloy, the fatigue damage is distributed in accordance with the ex-β grain texture, with occasionally microcracks coalescence favouring the initiation of the main crack. All cracks initiate at the α platelets-matrix interface.

Characteristics of fatigued NiTi and SiC coated materials

Results concerning the fatigue life of both treated titanium alloys are given in Table 2. Important improvements have been obtained with DIM coatings especially for the Ti6246 alloy. Indeed for this substrate, both NiTi and SiC coated samples have been cycled up to respectively 13 and 18 times the reference fatigue life without developing any damage at ±750 MPa. At the higher stress amplitude of

TABLE 2 – Number of cycles at failure.(* : stopped without surface damage.)

	$\Delta\sigma/2$ (MPa)	Coating	Thickness (μm)	N_f (cycles)
Ti 6Al 4V	750	uncoated	—	5,000
	750	SiC	0.25	10,800
	750	NiTi	0.35	5,140
	750	SiC	1.0	5,545
	750	NiTi	1.5	14,400
Ti 6246	750	uncoated	—	12,000
	750	SiC	0.2	160,865 *
	750	NiTi	0.2	216,000 *
	850	uncoated	—	3,900
	850	SiC	0.2	9,012
	850	NiTi	0.2	5,600
	850	NiTi	1	12,600

±850 MPa, the effects of both coatings are comparable to those obtained on Ti6Al4V at ±750 MPa : for thin films (~ 0.2 μm), significant improvements have been obtained with SiC (~ $N_f \times 2$) whereas no beneficial effect has been noted with NiTi films. For thicker coatings (1μm), results are inverted. Indeed, fatigue life improvements are important with NiTi (~ $N_f \times 3$) while no increase of the number of cycles at failure has been obtained with SiC. Results concerning SiC thick coating are quite different : a multicracking of the film appears of the beginning of fatigue tests due to the SiC brittle character (Figure 2). However cracks which initiate perpendicularly to the stress axis only concern the SiC coating without penetrating into the substrate.

In all the cases, excepted for SiC 1μm thick, most of the microcrack sites are suppressed. The number of macrocracks which form is frequently lowered to only one, sometimes accompagnied by few secondary microcracks instead of several hundreds on the reference untreated material. SEM observations through DIM thin coatings have shown that the crack initiation sites are similar to those encountered on uncoated materials (Figure 3).

DISCUSSION / CONCLUSIONS

We have shown that the DIM technique permits to engineer thin amorphous NiTi and SiC films which remain well adherent to both Ti6Al4V and Ti6246 alloys during cycling and leads to important improvements of their fatigue resistance.

NiTi coatings act as a barrier at the surface which modifies deformations and crack initiation in the substrate since only one or few cracks can form. The low Young's modulus of NiTi films and their high ductility are at the origin of these beneficial effects on crack initiation processes. Moreover, thicker is the film, greater is the barrier effect. Indeed, for thin films (~ 0.2 μm), the α platelets-matrix interface deformations lead to the shearing of the coating more easily.

In contrast, thick DIM SiC coatings are not so efficient for improving the fatigue resistance of titanium alloys. Indeed, their higher Young's modulus (235 GPa instead of ~ 120 GPa for titanium substrates) induces a high stress amplitude during cycling. Moreover their brittle character favours their breaking from the first cycles. The « breaking point » of these films being reached, the multicracking process which takes place ruins the expected beneficial effects. However, thin SiC films which are less brittle and which permit to delay crack initiation significantly can be cyclically deformed under the same conditions without reaching their « breaking point ». The better improvements obtained in this case compared to those corresponding to thin NiTi films has been attributed to a better resistance of SiC with regards to environment effects.

In all the cases, the fine and homogeneous Ti 6246 microstructure is more favourable to improve its fatigue life than the heterogeneous structure of Ti6Al4V alloy. In this latter alloy, the large slip lengths which appear easily within the colonies (300 μm) of aligned α grains initiate trans or intergranular α/α cracks and can lead to the shearing of the coatings. On the contrary the fine and homogeneous structure of the Ti 6246 is more resistant to crack initiation. Moreover the first microcracks do not appear before the half fatigue lifetime of the Ti6246 while they appear at the beginning of cycling for the Ti6Al4V (less than 10 % of N_f).

Then it is clear that beneficial effects can be obtained with very thin films when a good compromise is reached between the mechanical properties of the DIM films (low Young's modulus, ductile or brittle character...) and the deformation conditions imposed to the coating during the fatigue tests through the deformation mode of the substrate (total longitudinal deformation, shearing at the interface due to localization effects in the bulk).

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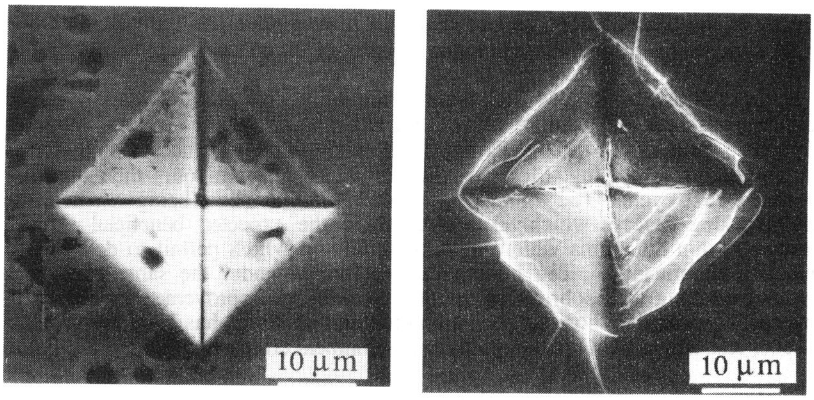

NiTi

SiC

Figure 1 S.E.M. observations of NiTi (1.5 μm) and SiC (1 μm) coating deformations after equivalent micro-Vickers indentation.

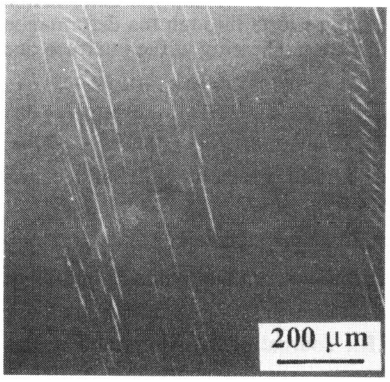

Figure 2 Surface aspect at failure of a SiC (1μm) coatedTi6246.

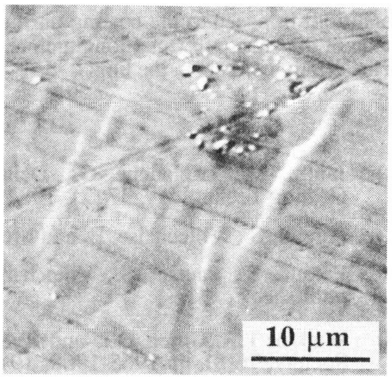

Figure 3 Crack initiation site on a NiTi (0.2μm)coated Ti6246 sample.