Mechanical Properties of Triclosan Containing Sol-gel Thin Films on Titanium Alloy

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Statement of Purpose: Post-surgical infections are one of the major factors that affect hospital stay duration and associated medical expenditures in the United States. Fraction fixation materials used to set fractures are susceptible to baceterial biofilm formation and infection [1]. Thus, surface engineering approaches are being considered to to address these issues. Antibiotic containing sol-gel thin films have been coated on titanium alloy fracture fixation material with the goal to prevent biofilm formation and infection [2]. Thin films bonded on metallic surfaces are usually in a state of residual tension. Therefore, thin films can fail under load bearing conditions. Herein we determine the interfacial adhesion properties of sol-gel thin films on titanium alloy substrates.

Methods: Ti6Al4V samples, 0.5mm thick, 25mm long and 12mm wide, were used for the coating deposition. Two different substrates were prepared: polished with sandpaper (SiC, 800/2400 grit); or, sandblasted with 50µm Al_2O_3 under 40 psi. The samples were cleaned, rinsed with deionized water and passivated in 35% nitric acid for 1 h. Triclosan solutions in ethanol (100 mg/ml) were used for incorporation of the bactericidal triclosan during the sol synthesis. TEOS (tetraethoxysilane), ethanol, deionized water, and 1N HCl were mixed to form an acid-catalyzed silica sol. Sol-gel coatings containing 0% (SG) and 10 wt% triclosan (SGT) were used in the study. The dipping-deposition of the sol-gel films was performed as described elsewhere at a withdrawal speed of 300 mm/min [2]. Samples were pulled uniaxially at a crosshead speed of 0.5 mm/min in an Instron universal mechanical testing machine. The samples were strained stepwise with small increments from 1% to 15% (1%, 3%, 7%, 13% and 15%). Samples were examined under SEM to determine the strains at onset and saturation of cracking. The Young's modulus (E) and Hardness (H) were derived from nano-indentation tests.

The film fracture energy (γ_f) was calculated by the following equation 1 [3]:

$$\gamma_f = \frac{\sigma_c^2 t}{E_f}\left[\pi F(\alpha_D) + \frac{\alpha_c}{\sqrt{3}\alpha_Y}\right]$$

Where $F(\alpha_D)$ is a function of the elastic modulus mismatch between film and substrate, and α_Y is the yield stress of the substrate. α_c is the critical stress for film cracking which was determined from equation 2:

$$\sigma_c = \varepsilon_c E_f$$

where ε_c is the strain to onset of cracking and E_f is the film's Young's modulus. Also, the interfacial fracture energy was calculated using equation 3:

$$\gamma_i = E_f t \varepsilon_i^2 / 2$$

Where *t* is the thickness of the film, which is 200 nm for the sol-gel films of this study. The ε_i is the strain at the crack saturation of the film.

Results: The Young's modulus calculated from the nanoindentation test for films with and without triclosan was 66.05±9.04 GPa and 61.02±4.39GPa respectively.

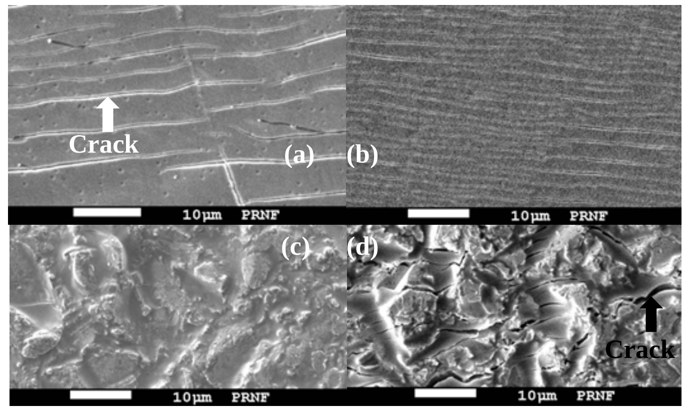

Figure 1 SEM of the typical crack pattern: a) polished, strained at ε = 1%. b) polished, strained at ε = 3%. c)sandblasted, strained at ε = 7%. d) sandblasted, strained at ε = 13%.

The cracks formed transversely in the brittle ceramic coating layer, albeit more independent from the tensile direction on sandblasted surfaces (figure 1). Cracking occured when the strain exceeded 1% and 7% for polished and sandblasted substrates respectively. The crack density increased with higher strain, and reached a steady state at a strain of 3% and 13% for polished and sandblasted substrates respectively (Table 1). After substituting all parameters into equations 1 and 3, we found the film fracture energy and interfacial fracture energy for films on sandblasted substrates with and without triclosan were 267.18J/m^2, 103.12 J/m^2; 303.82J/m^2 and 111.62J/m^2. These values were much higher than those for films on polished substrates (2.59J/m^2, 5.49J/m^2; 2.84J/m^2, 5.94J/m^2, respectively).

Table 1 Mechanical properties and related film parameters

Sol-gel film		Crack onset strain	Crack saturation strain	Crack spacing (µm)	Young's modulus, E(GPa)
Polished	SG	1%	3%	2.02±0.12	61.02±4.39
	SGT	1%	3%	2.22±0.09	66.05±9.04
Sandblasted	SG	7%	13%	4.69±0.13	/
	SGT	7%	13%	4.76±0.14	/

Conclusions: Mechanical properties of sol-gel films containing a bactericidal molecule or not on titanium alloy substrates were measured. Adding triclosan to the sol-gel film has no measurable effect on the mechanical properties. However, the interfacial fracture energy and film fracture energy were greatly affected by the substrate conditions. The sol-gel films on sandblasted substrates have a much higher adhesion strength.

References: [1]Subra. K. Intel J O & M Impl. 2009;24; 4:616 -626. [2] Shula R. Biomater. 2007;28:1721-1729. [3] Atanacio AJ, Latella BA, et al. Surf & Coat Tech 2005;192:354– 364