Bending Fatigue Properties of Nanoprocessed CP Titanium Spine Rods

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INTRODUCTION: Various advanced processing methods have been devised to produce nanoprocessed biomaterials. One recent development known as Equal Channel Angular Pressing (ECAP) has been described elsewhere (Valiev R.Z., Proc Mat & Proc for Med Dev Conf, 2004, 362-367) for producing nanostructured metallic biomaterials. The present study evaluated the bending fatigue properties of ECAP processed Commercially Pure (CP) titanium (Ti) Grade 4 spine rods as part of an on going research project which includes static bending, tensile, corrosion, and microstructure characterization.

MATERIALS AND METHODS: Annealed 25.4mm diameter Ti Grade 4 bar stock, heat no. H4359, was selected for the ECAP trial and met the following analysis: C=0.052%, N=0.005%, O=0.34%, H=0.0015%, Fe=0.02%, Ti=balance. The starting bar stock was certified to the mechanical properties in ASTM F 67. Bar specimens were ECAP processed by Metallicum LLC, Santa Fe, New Mexico, and centerless ground to a finish size of 8 mm diameter. The nanoprocessed material had an ultra-fine grained microstructure with an ASTM grain size of 25. ASTM F 67 control specimens consisted of cold worked 8 mm diameter Ti Grade 4, heat no. R3018, with 0.006%C, 0.006%N, 0.35%O, balance Ti. A grain size of ASTM 9.5 was measured for the control specimens. All samples were bead blasted with Zirshot Z-600 ceramic bead blast media and electrochemically anodized in a sulfuric acid bath to provide a gold anodized film. Thickness of the gold anodized film was approximately 140 nanometers. All surface treatments represented finishing operations standardized by Synthes for spinal rods.

Four-point bending fatigue testing was performed according to ASTM F 2193 at 10Hz frequency, in air, room temperature, R=0.1, with a modified four-point

roller spacing to accommodate the 120 mm bar lengths. Maximum compressive load versus cycles to failure was measured in duplicate for each selected bending fatigue load. Runout (absence of fatigue failure) was established as 5 million cycles to failure. Scanning Electron Microscopy (SEM) analysis was used to examine failed specimens representing the low-cycle and high-cycle fatigue regime. High-cycle fatigue is defined as greater than 100,000 cycles to failure.

RESULTS AND **DISCUSSION:** The four-point bending fatigue curve is shown in Figure 1 (solid line = control; dashed line =ECAP). The control samples achieved runout within a compressive load range of 2300-2465 N while the runout for ECAP rods was between 1725-2156N. The fatigue resistance of the

ECAP spine rods was dramatically inferior to the control specimens.

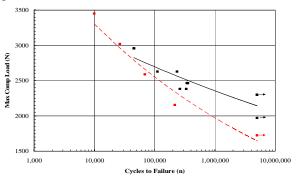


Figure 1. Bending Fatigue Curve

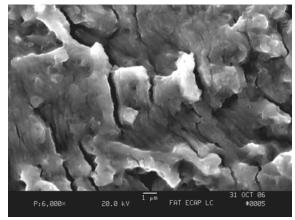


Figure 2. Striations on a ECAP Low Cycle Sample

Examination of the fracture surfaces of both the cold worked Grade 4 CP Ti and the ECAP subjected to higher loads with the fracture in the low cycle range (<10⁵ cycles) showed significant differences in fracture surface morphology. Both materials showed striation spacing in the 0.3 to 0.5µm range (Figure 2). The ECAP material showed a larger and less ductile area of overload fracture. The ECAP CP Ti subjected to lower loads resulting in fracture in the high cycle region (>10⁵ cycles) also exhibited a greater area of overload fracture than did the cold worked CP Ti Grade 4. Further investigations of the fracture mechanisms are in progress.

CONCLUSIONS

- 1. Dynamic testing of ECAP CP Ti grade 4 spine rods indicated inferior four-point bending fatigue properties when compared with conventionally cold worked spine rods.
- 2. SEM surface examination of failed ECAP specimens showed significant differences in fracture morphology compared to conventionally cold worked CP Ti Grade 4.