Journal of the Mechanical Behavior of Biomedical Materials 83 (2018) 94–101

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm

Random spectrum fatigue performance of severely plastically deformed titanium for implant dentistry applications

D. Rittel[a,*], K. Shemtov-Yona[a], R. Lapovok[b]

[a] Faculty of Mechanical Engineering, Technion, 32000 Haifa, Israel
[b] Institute for Frontier Materials, Deakin University, Australia

ARTICLE INFO

Keywords:
Severe plastic deformation
Titanium
Dental implants
Random spectrum
Fatigue

ABSTRACT

Severe plastic deformation (SPD) has long been known to confer superior mechanical properties for many metals and alloys. In the general field of biomedical devices, and dental implants in particular, the superior strength of SPD-processed commercially pure (CP) titanium, that may surpass that of the stronger Ti6Al4V alloy, has been associated with a superior fatigue resistance. Such a property would make those materials both biocompatible and strong alternatives to the currently used titanium alloy.

However, the fatigue characterization reported so far in the literature relies on a very small sample size, thereby precluding any meaningful statistical analysis.

This paper reports and compares systematic fatigue testing of various grades as-received and SPD processed Grade 4 CP-Ti using the recently developed random spectrum loading approach, in both air and 0.9% saline solution.

The results of this study do not support the claim that the SPD process, albeit causing noticeable strengthening, confers any advantage to Grade 4 CP-Ti in terms of fatigue response.

1. Introduction

With the discovery of the Hall-Petch relationship (Hall, 1951; Petch, 1953), a substantial research effort has been invested in producing and characterizing the mechanical properties of “small-grained” or simply nanograined materials. The main incentive for this effort is the substantial increase in yield (and flow) strength of ultrafine grained materials, albeit at the expense of their strain hardening capabilities (Jia et al., 2001; Ramesh, 2009; Rittel et al., 2017b), or even their propensity for dynamic shear localization under impact, see. e.g. (Jia et al., 2003; Wei et al., 2004). Yet, the benefits of the grain refinement are not experienced for any grain size, but there exists a limit for which the “inverse Hall-Petch” operates, causing a reduction in strength due to grain boundary sliding as opposed to pure dislocation-mediated plasticity (Mercier et al., 2007; Trelewicz et al., 2008).

The production of nanograined materials involves a “break up” of the metallic grain structure while limiting recrystallization and grain growth phenomena. The most popular method of achieving a very fine grain size consists of the application of very large strains (severe plastic deformation – SPD) using ECAP (equal channel angular pressing), or other well developed techniques (Valiev et al., 2006). ECAP processing involves repeated deformation through the die with two equal intersecting channels. The severe shear strain is introduced in the narrow band along channels intersection without change of the rod geometry. 90° rotation of the sample between passes, known as route B_C, results in e intensified grain fragmentation (Valiev et al., 2006).

Commercially pure (CP) titanium and Ti6Al4V are very often used for the fabrication of prosthetic and dental implants, for their combined strength and biocompatibility (Brunette et al., 2015; Elias et al., 2008). Concerning dental implants specifically, the latter may experience relatively rare mechanical failures due to fatigue that develops with the repeated mastication loads in variable intraoral environment (K Shemtov-Yona and Rittel, 2016a; Shemtov-Yona and Rittel, 2015, 2014). Given the relative variability of the mastication loads, one would naturally seek to develop/use stronger alloys in implant dentistry (Rittel et al., 2017a). Here one must note that, whereas CP Ti is universally recognized as a biocompatible material, the Ti6Al4V alloy has raised concerns regarding the potential toxicity of its alloying elements (Elias et al., 2008; Matusiewicz, 2014).

However, it is also well evidenced that Ti6Al4V is noticeably stronger than CP-Ti, a property that makes the former quite attractive for prosthetic applications.

The fatigue properties of metallic alloys are usually reported in terms of load vs. number of cycles to failure (S/N curve (Suresh, 1998)),

---

* Corresponding author.
E-mail address: merittel@technion.ac.il (D. Rittel).

https://doi.org/10.1016/j.jmbbm.2018.04.012
Received 26 February 2018; Received in revised form 29 March 2018; Accepted 12 April 2018

noting that the latter may require extensive statistical processing (Nicholas, 2003; Schijve, 2009; Shemtov-Yona and Rittel, 2016b). Without detailed comparative analysis, based on the testing of a large sample size, one cannot draw firmly anchored conclusions regarding ranking of various grades of material. Concerning dental implants, the current standards (ISO, 2016) require a rather small sample size per load level that precludes any statistical analysis.

Several studies have established the fact that severe plastic deformation can significantly increase the yield and flow strength of CP-Ti, bringing those properties close or even superior to those of Ti6Al4V, see e.g. (Elias et al., 2008; Medvedev et al., 2015, 2016a; Mishnaevsky et al., 2014; Valiev et al., 2006).

In parallel, a few studies have compared the fatigue properties of those materials, and come to the conclusion that nanograined CP-Ti has superior fatigue properties when compared to Ti6Al4V (Figueiredo et al., 2014; A. Medvedev et al., 2016b; Medvedev et al., 2015). However, the tested sample size was relatively small (one specimen per load level), so that this conclusion was qualitative and not statistically supported.

In the field of fatigue testing of dental implants, it was recently proposed to address the problem by means of functional testing of the implant. The idea here is that instead of looking for a fatigue limit, one should expose the implant to a random loading spectrum until its final fracture (K Shemtov-Yona and Rittel, 2016b; Shemtov-Yona and Rittel, 20162016c). One of the attracting characteristics of this approach is that each tested group is characterized by its mean longevity with its standard deviation, making the comparison between different tested groups quite straightforward. This comes as an alternative to the S/N curve for which each load level has its mean longevity and standard deviation, together with the complexity that in the finite life regime, the number of cycles to failure is statistically distributed, whereas in the transition regime, the load becomes statistically distributed.

Consequently, this paper compares the fatigue response of SPD CP-Ti with that of as-received Ti6Al4V, using the random spectrum loading approach, for both room-air and saline solution testing.

The paper is organized as follows. We first describe the materials, specimens and briefly the random spectrum procedure. Next we report the results obtained for each batch of tests, followed by a discussion in which the results are statistically compared, ending by a conclusions section.

2. Materials and methods

2.1. Materials

The tested materials consist of undeformed and SPD processed Grade 4 CP-Ti, and as-received Ti6Al4V, (12.7 mm rod). All materials, except for the Ti6Al4V, were kindly provided by Dr. R. Lapovok. Table 1 lists the designation of the materials, noting that the SPD processed materials underwent 4 passes of ECAP at 2 mm/s speed and a variable amount of additional cold work (drawing).

Table 1
The materials tested in this study. Note that 4P360-1 and 2 underwent similar processing but originate from 2 different batches.

Original Marking	Designation	Material	Remarks
Round 12.7	AR	CP Ti	Undeformed-reference
Round 5	4P480	CP Ti	4 passes + drawing, total strain 480%
Al 0017-23	4P360-1	CP Ti	4 passes + drawing, total strain 360%
4 passes	4P360-2	CP Ti	4 passes + drawing, total strain 360%
Ti6Al4V	Ti64	Ti6Al4V	As-received

2.2. Specimens

The specimens used in both static and fatigue tests were cylindrical, 20 mm long with a nominal diameter of 4.85 mm. A square notch was machined at mid-length, 1 mm in width and nominal depth of 0.25 mm. The Appendix section provides details about each tested specimen, including its exact dimensions, as a result of slight variability in the manufacturing process.

2.3. Random spectrum

The random spectrum loading methodology has been described in detail in (K Shemtov-Yona and Rittel, 2016b; Shemtov-Yona and Rittel, 20162016c), and will only be briefly exposed here. The idea is to generate a random loading spectrum that spans from 10 N to 2500 N in this work, generated as a series of loading blocks, with a randomly variable frequency of 1–3 Hz. Some of the blocks are randomly assigned a 0 load value to mimic pauses (relevant for testing in fluid atmosphere). The upper limit is decided upon after quasi-static testing and all the reported results in this work are generated upon application of the same spectrum. The random spectrum approach is described schematically in Appendix 1. Consequently, the outcome of the test is the time to fracture of the specimen, as reported in the Appendix 2 section.

2.4. Loading apparatus and setup

The loading apparatus is an MTS servo-hydraulic machine, driven under load control. The specimen is inserted in a holding block, with the bottom flank of the notch aligned flush with the block, thereby creating the initial stress concentration required to generate a fatigue crack in a controlled location, namely the small square notch. All the specimens are inclined at a 30 degrees angle with respect to the machine crosshead. The configuration used here is identical to that used in all our previous works, see e.g. (Shemtov-Yona and Rittel, 2016a).

Tests were carried out in room air or in 0.9% saline solution, as in (K. Shemtov-Yona and Rittel, 20162016c), but the static tests were only carried out in room air since the saline solution is not expected to influence the mechanical characteristics of the material over short time durations that are characteristic of quasi-static testing.

2.5. Fractographic analysis

All specimens were examined visually. Next, four representative specimens, all made of CP-Ti were selected for fractographic examination using a Quanta 200 scanning electron microscope.

3. Results

3.1. Quasi-static testing in room air

Two specimens of each material were tested, as shown in Table 2.

The results of the quasi-static bending tests clearly indicate 2 kinds of mechanical characteristics: The “soft” material, CP-Ti AR, for which the failure load is inferior to 4000 N, and the remaining “hard” materials, 4P480, 4P360-1 and 2, and Ti6Al4V, for which the failure load clearly exceeds 5000 N. At this stage, we did not carry out additional

Table 2
Quasi-static test results.

Designation	Material	Failure load 1 [N]	Failure load 2 [N]
AR	Gr 4 CP Ti	3836	3873
4P480	Gr 4 CP Ti	5429	5880
4P360-1	Gr 4 CP Ti	5354	5295
4P360-2	Gr 4 CP Ti	5391	5273
Ti64	As-rec. Ti6Al4V	5450	5367