Fatigue behaviour of CG and UFG titanium: DIC and fractography studies

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Abstract. The manuscript is devoted to the investigation of fatigue behaviour of course-grain (CG) and ultrafine-grain (UFG) titanium grade 2 by means of digital image correlation (DIC) and scanning electron microscopic fractography. The UFG state has been obtained utilizing severe plastic deformation technique. The results show that the high amount of defects (grain boundaries, dislocations, etc.) in UFG suppress plastic deformation and leads to earlier crack initiation, however at the macrocrack growth stage these impurities are obstacles for transgranular mechanism of fracture.

1. Introduction

The commercially pure titanium grade 1-4 is the most widely used material for dental and orthopaedic implants due to its biocompatibility, cost and relatively good processability [1]. However one of the very important limitations in a case of highly loaded prostheses is low mechanical properties which do not allow weight reduction of the components made from titanium. The prospective technique for enhancement of the mechanical properties is severe plastic deformation [2,3] which results in grain refinement and subsequent increase in yield and ultimate stresses [4,5] of the material, but due to lack of plasticity it could reduce of fatigue durability [6] which is also important for long-used implants.

Present study is aimed at investigation of fatigue properties and fracture kinetics of ultra-fine grain titanium (grade 2) in comparison with initial course-grain state.

2. Materials and fatigue test conditions

2.1. Grain refinement procedure

For the grain refinement the severe plastic deformation technique was implemented which consisted in three stages. At the first stage the blanks were subjected to abc-pressing and under elevated temperature (400-500°C) at the strain rate of $10^{-3} - 10^{-1}$ s⁻¹. Strains at each pressing cycle were 40-45%. At the second stage the blanks were deformed using rolling with the total strains about 90%. The third stage was annealing at $T=350°C$ for t=1 hour aimed at decreasing internal stresses caused by severe deformation. The final blanks had the shape of narrow thin bands with dimensions of $1.3 \times 5 \times 70$ mm (thickness × width × length).

The average grain size of the course-grain titanium is $25 \mu$m, while it is $0.2 \mu$m for ultrafine-grain titanium.
2.2. Estimations of crack length and maximum strains at the crack tip

The coordinates of crack tip position were estimated based on the images of the specimen surface at different operation time. Of course, the accuracy of such technique is lower than SEM based measurements, but comparable or higher than evaluations made by compliance method (COD gauge at the specimen edge).

The next level of measurement was performed via DIC – computation of displacement and strain fields based on the specimen images. The non-uniform surface of the polished titanium played the role of a speckle pattern which is obligatory for DIC. Then the extensometer tool was used in order to perform the evaluation of strain values directly at the crack tip which position has been found earlier. The extensometer length was 50 px which corresponds to ~30 µm. So the precise measurements of the strain response of the crack tip on the applied load during the fatigue tests were obtained.

2.3. Fatigue test specimens and loads

As far as the final titanium pieces after refinement were small therefore for the fatigue tests were cut the miniature specimens with single edge notches for crack initiation (figure 1). These specimens made of two types of materials – CG and UFG titanium – were subjected to cyclic loading under maximum load \( P_{\text{max}} = 0.6 \, \text{kN} \) and loading ratio \( R = 0.1 \) which corresponds to maximum stress intensity factor (SIF) \( K_{\text{max}} \approx 16 \, \text{MPa} \cdot \sqrt{\text{m}} \) and SIF range \( \Delta K \approx 14.7 \, \text{MPa} \) at the beginning of the test.

![Figure 1. Fatigue specimen shape and dimensions.](image)

The image acquisition of the crack tip at high magnification was performed under fixed maximum load \( (P_{\text{max}}) \) every 2000 cycles before crack initiation and every 500 cycles during the stage of macro crack growth. The digital camera follows the growing crack thus the tip was always in area of interest.

3. Experimental results and discussion

As a result of fatigue test the average fatigue durability was 210000±28000 cycles for CG titanium and 180000±38000 cycles for UFG titanium specimens.

3.1. DIC study

The crack growth process could be divided into two stages: (1) crack nucleation and (2) micro crack propagation. The durations (figure 2a) of the first stage were 186000 (88% of life time) and 135000 (77% of life time) cycles for CG and UFG correspondingly whilst the second one – 25000 (12%) and 41000 (23%). The crack growth stage for two specimen types is illustrated in figure 2b as a dependence on the life time. It could be noted that the crack appearance in UFG titanium occurs earlier than in CG Ti, whereas the total crack length achieved before fracture of UFG Ti is much higher for all tested specimens (approximately 2.5 mm for CG and 3 mm for UFG).

Figure 3a demonstrates fatigue fracture kinetic diagrams; it is seen that when crack is small the propagation rates are near the same, but when crack reaches ~0.7 mm (\( \Delta K \approx 20 \, \text{MPa} \cdot \sqrt{\text{m}} \)) it leads to the higher growth rate of the crack in CG titanium. However at the end of the test the rates became close to each other again, so the middle regions of the fracture diagrams of titanium are sensitive to the grain size.

Traditionally the processes of crack growth could be attributed to two main factors: threshold range of stress intensity factor (\( \Delta K_{\text{th}} \)) and effective range of stress intensity factor (\( \Delta K_{\text{eff}} \)) – both of them are
different for presented types of specimen due to different yield stress which manages the near-tip stresses ($\sigma^*$). The effect of each parameter needs to be studied.

![Figure 2](image1.png)

**Figure 2.** Bar diagram of crack nucleation and propagation stages (a) and the dependences of total crack length on the specimen life time (b) for course-grain and ultrafine-grain titanium.

Evolution of strains at the crack is shown in figure 3b and they demonstrate nonlinear behaviour. The last measurement point (prior to fracture) for UFG titanium is not presented in the plot due to high deformations and the loops of correlation between two adjacent images.

![Figure 3](image2.png)

**Figure 3.** Fatigue fracture kinetic diagrams (a) and change of local strains at the crack tip during crack propagation (b) for course-grain and ultrafine-grain titanium.

3.2. **SEM fractographic study**

The SEM images of fracture surface of CG titanium are presented in figure 4. It demonstrates the panoramic view and high magnification images (2000x) of different fracture textures at different crack length. The fracture pattern consists of three texture types representing different crack propagation processes – origin area, where micro- and meso- cracks have been merged in one micro crack going thought the whole specimen thickness; area of stable crack growth and static fracture area at the right edge of the specimen. It should be noted that the during stable crack propagation period the texture becomes more rough and more cracks seen (figure 4b and 4c). Static fracture zone has a mostly uniform
pattern of ductile fracture. Moreover this area was significantly narrowed due to high level of plastic deformation.

Figure 4. Panoramic image (a) of fatigue fracture surface for CG titanium specimens and surface texture at different crack length (b,c,d).

SEM images of UFG titanium fracture surfaces (figure 5) demonstrate more brittle fracture processes but still it consists of three main zones. The stable crack growth stage is represented by very smooth and fine microstructure with no river-like pattern (figure 5a). However, the micro- and mesocracking also occurs at longer crack length and it is even more pronounced as for CG titanium (figure 5b). Prior to the final fracture the crack extension at individual cycles is seen on the surface as a separate bands divided by striations (figure 5c).

Figure 5. Panoramic image (a) of fatigue fracture surface for UFG titanium specimens and surface texture at different crack length (b,c,d).
4. Conclusion
The paper presents the comparison of fatigue fracture kinetics of CG and UFG titanium specimens by means of digital image correlation technique and SEM fractography. Analysis of the results obtained leads to the following conclusions:

- at the crack nucleation stage presence of many grain boundaries in UFG Ti prevent development of plastic deformations which would reduce local stresses and accumulated elastic energy in in the vicinity of appeared microcracks, thus crack emerges on the surface of UFG Ti earlier than for CG Ti;
- at the crack propagation stage UFG Ti has lower crack growth rates due to grain boundaries and dislocations which in this case are obstacles for transgranular type of fracture.

Therefore a lot of structural defects in UFG Ti could have a negative effect on crack initiation at the first stage and then have positive impact by suppression of crack growth processes at the second stage.

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