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FATIGUE PROPERTIES OF NANOCRYSTALLINE TITANIUM

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Abstract. Fatigue resistance of nanocrystalline commercial pure titanium obtained by hydrostatic extrusion was investigated. Coarse grained titanium and Ti6Al4V alloy were used as the reference materials. The fatigue limit and fatigue life examined at constant stress amplitude, were found to increase significantly compared to those of the coarse-grained counterparts. It is suggested that the observed improvement of the fatigue performance results from the microplasticity being suppressed by the microstructure refinement.

1. INTRODUCTION

Bulk nanocrystalline (NC) and ultrafine-grained (UFG) materials produced via severe plastic deformation are currently developed for a number of engineering applications. Hydrostatic extrusion (HE) is one of the methods which allow producing severe deformation in bulk samples without fracture and yields grain refinement and significant hardening.

Titanium is widely used for medical applications owing to its low weight and very good biocompatibility, especially the ability of osseointegration. However, the application of titanium is limited by its poor wear resistance, relatively low tensile strength and fatigue limit. The fatigue of Ti policrystals with conventional grain sizes has been investigated in the past. It has been shown that the fatigue limit increases significantly with the grain refinement from 100 to 9 μm. This study reports on fatigue resistance of nanocrystalline Ti-Grade 2 (NC) with emphasis on its microstructural aspects. It broadens our knowledge about the mechanical properties of nanocrystalline titanium under the conditions of dynamic loads [1-4].

2. EXPERIMENTAL PROCEDURES

The material examined in the study was commercially pure titanium subjected to HE at room temperature. The extrusion process was performed at the Institute of High Pressure Physics, Polish Academy of Sciences in Warsaw under the project coordinated by Faculty of Materials Science and Engineering of Warsaw University of Technology. The samples in the form of cylindrical rods were extruded to reduce the diameter 33 mm to 5 mm. The hydrostatic extrusion technique was described in detail in our earlier publications [5-9]. The microstructure of titanium processed by HE was examined by scanning and transmission microscopy (SEM/STEM Hitachi S 5500) and using a computer image analy-

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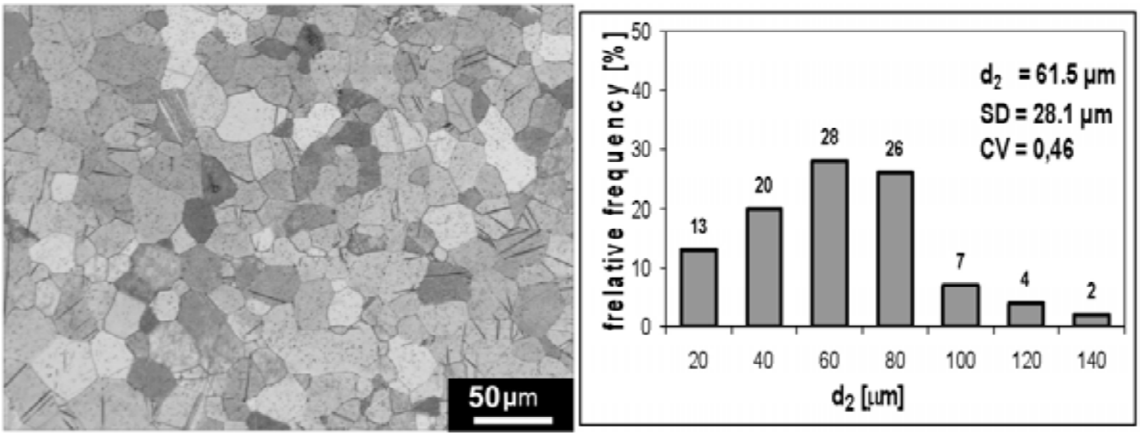

Fig. 1. Microstructure of CG Ti Grade2 in the as-received state.

sis. The mechanical properties of the investigated materials were characterized by measuring the microhardness ($HV_{0.2}$) and fatigue resistance. The fracture surface was observed using scanning microscope HITACHI S-2600N. The references materials were coarse grained titanium (CG) with a grain size of 20 μm and Ti6Al4V alloy.

3. RESULTS AND DISCUSSION

Fig. 1 is a TEM micrograph showing the microstructure of the titanium sample after multi-stage extrusion at true strain of 3.8 (Fig. 2). A significant grain refinement is evident when the microstructure is compared with the initial microstructure shown in Fig. 1. The as–received CG Ti has a coarse grained recrystallized structure with the average grain size d_2 = 20 μm in both sections. The microstructure of the Ti6Al4V alloy, the other reference material, contained of equiaxial alpha phase grains and beta phase grains of irregular shape located at the "triple point" of the alpha grains (Fig. 3). The average grain size of the alpha phase was about 5 μm.

3.1. Fatigue properties

The fatigue testing was performed using the servo-hydraulic testing machine MTS 858. The specimens were cylindrical and "dog-bone" in shape. After mechanical polishing, the specimens were subjected to tension-tension (R=0,1) loading at a frequency of 10.0 Hz with a sinusoidal waveform in the air environment. Several samples of each of the NC and CG materials were tested to failure at a given stress range, and the number of cycles to failure was recorded.

The effects of the grain size on the fatigue resistance of tested materials are plotted in Fig. 4 in the

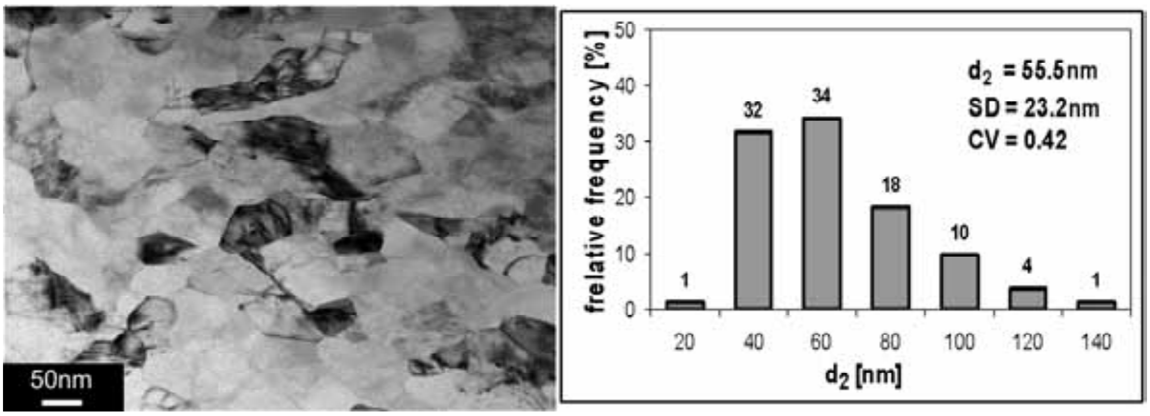

Fig. 2. Microstructures of NC Ti Grade2 after HE.

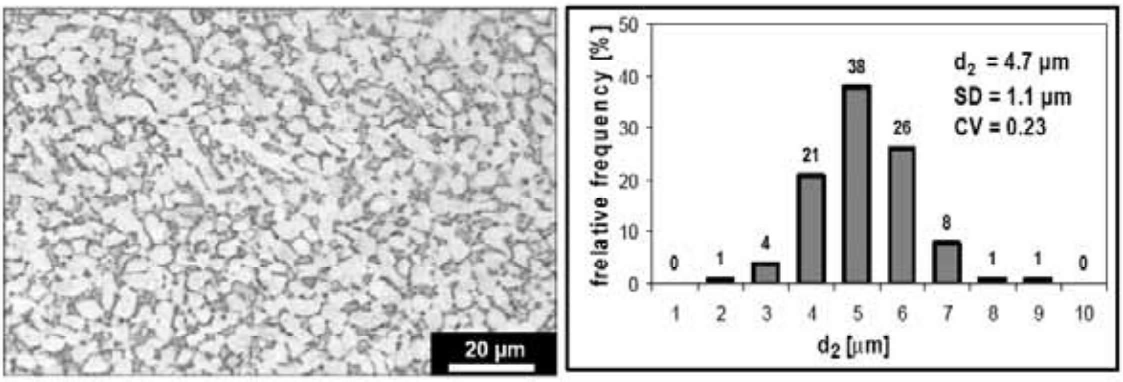

Fig. 3. Microstructures of the Ti6Al4V alloy and the grain size of a phase distribution.

form of the stress–life (S–N) diagram. It can be seen that the NC Ti with an average grain size of approximately 60 nm has a much greater resistance to stress-controlled fatigue than the CG Ti, but lower than Ti6Al4V alloy. The results shown in Fig. 4 clearly illustrate that a grain refinement of pure Ti leads to an enhancement in the resistance to S–N fatigue but the effect of the grain refinement is weaker then alloying effect.

3.2. Fracture surface analysis

Observations of the fatigue fractures of the samples permitted investigating how the microstructure refinement affected the fractographic features of their fatigue fracture. All the fractures show regions characteristic of fatigue cracking, namely the crack origin, fatigue crack development and final rupture (Figs. 5-7).

Among the materials examined, it was CG Ti whose fracture surface was most developed (Fig.

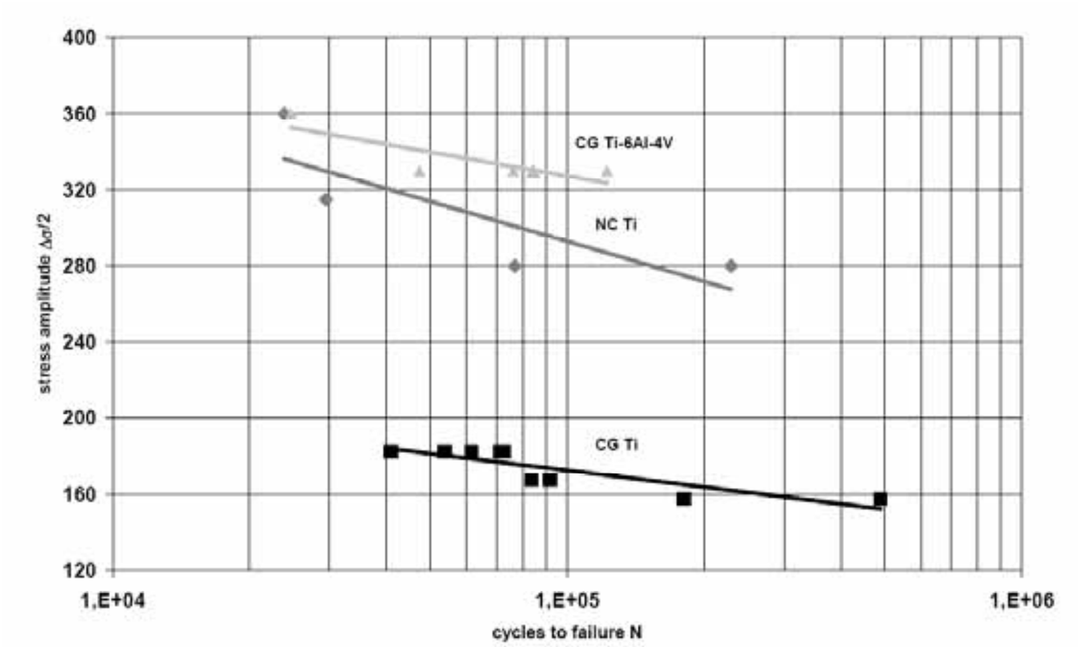

Fig. 4. A comparison of the S–N fatigue response showing the stress range versus number of cycles to failure for the NC Ti, CG Ti and Ti-Al-V alloy.

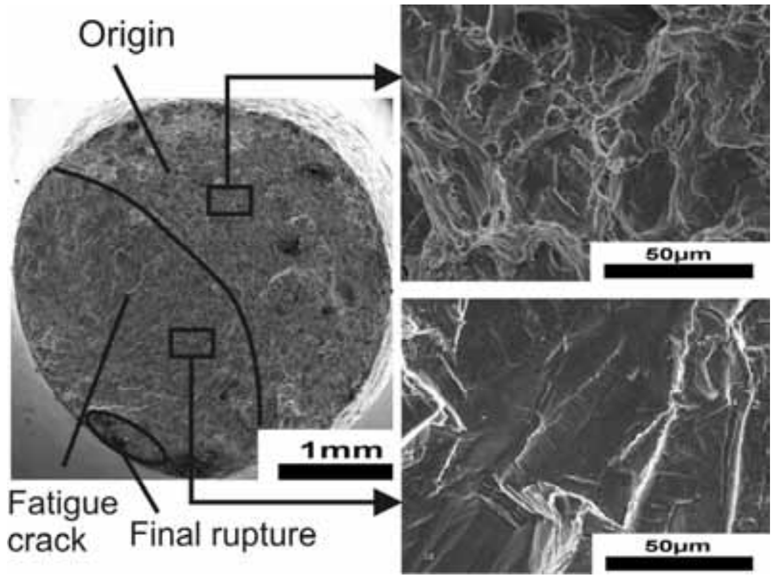

Fig. 5. Scanning electron fractograph showing the fatigue fracture features of CG Ti Grade2 in the as-received state.

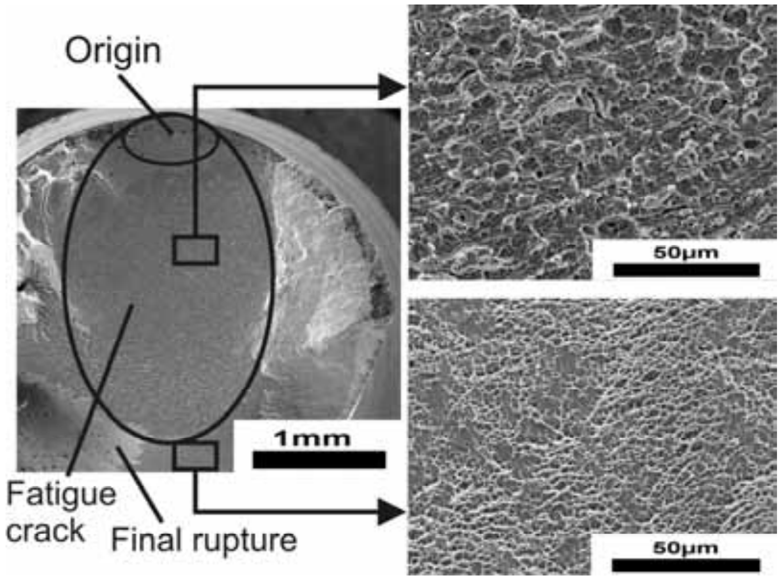

Fig. 6. Scanning electron fractograph showing the fatigue fracture features of NC Ti Grade2 after HE.

5). Observations at greater magnifications revealed the presence of facets of cleavage cracking, characteristic of materials with a compact hexagonal structure. The size of these facets was close to the average grain size, which suggests that the cracks propagated along the grain boundaries. We can also see fatigue lines present on the fractures. The fatigue fracture of CG Ti did not show microcracks. In NC Ti obtained after HE the fatigue fracture surface was smoother compared to that of the starting material (Fig. 6). A macroscopic photograph of this fracture showed traces of the propagation of the fatigue crack. The fracture through the fatigue crack development region had a mixed character (brittle-plastic) with numerous cracks with the length from several to several dozens of μm. The region of final rupture in this material had a pitting morphology, which is evidence that, under dynamically varying load, NC Ti behaved more plastically than CG Ti.

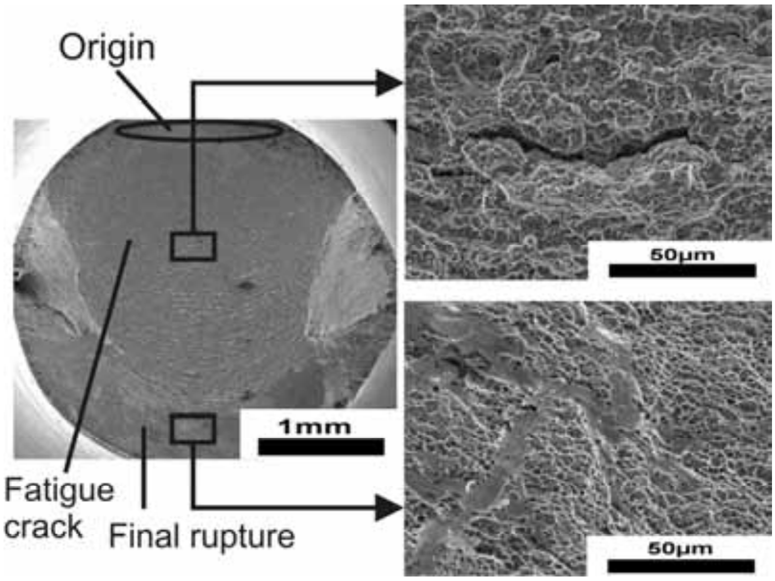

Fig. 7. Scanning electron fractograph showing the fatigue fracture features of Ti6Al4V alloy.

The morphology of the Ti6Al4V alloy was similar to that of NC Ti. In the fracture plane, the fatigue crack development region contained cracks several μm long and very numerous shorter (a few to several dozen of μm) cracks, which suggests that the fatigue cracking occurred here via joining and development of microcracks.

4. CONCLUSIONS

The study was concerned with the fatigue resistance of NC Ti produced by hydrostatic extrusion. The reference materials were CG Ti and the Ti6Al4V. The nanocrystalline titanium obtained by hydrostatic extrusion shows an increased limited fatigue strength, which is however lower than that of the Ti6Al4V alloy. The HE-induced grain refinement of titanium affected the fractographic features of its fatigue fracture. Thanks to the strongly refined microstructure, the fracture surface of nanocrystalline titanium was smoother than that of the microcrystalline material. The microstructure within the region of final rupture had a pitting morphology, which is evidence of a plastic character of the rupture.

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