Corrosion and Low Cycle Fatigue Behaviour of Surface Modified Ti-13Nb-13Zr Alloy

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By

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CHAPTER 8

Summary and Suggestions for Future Work

8.1 INTRODUCTION

This chapter of the thesis highlights the key findings and makes recommendations for further study.

8.2 SUMMARY

Detailed study was carried out on the near-β Ti-13Nb-13Zr alloy with respect to its microstructure, elastic modulus, and mechanical properties in different heat-treated conditions. Ultrasonic shot peening technique was used to modify the surface microstructure of the samples, solution treated at 900°C and water quenched having ~196 µm size grains to nanoscale of the selected heat-treated samples. There after a systematic study was conducted on the surface microstructural modification, electrochemical corrosion, static corrosion, low cycle fatigue behaviour and bio-activity of the Ti-13Nb-13Zr alloy. The study related to corrosion behaviour and bio-compatibility has been conducted on samples USSP treated for lower duration (15 to 120 seconds) as both of these properties are surface dependent and much higher duration of USSP treatment will severely damage the surface and adversely affect the corrosion and cellular response. In study related to fatigue behaviour of samples higher duration of USSP was taken for developing greater depth of modified layer that will prevent crack initiation and thereby helps in improving the fatigue life. Samples that were USSP treated showed grain refinement as well increase in residual stress at surface. These samples were stress relieved to see the effect of only grain refinement on the biocompatibility and fatigue behaviour. LCF behaviour of the Ti-13Nb-13Zr alloy in the Un-USSP, USSP, and USSP-stress relieved conditions (400 °C for 1 hour) was studied at room temperature at different total strain amplitudes and a constant strain rate of 0.005 s^{-1}. The important observations made on various aspects are listed at the end of the respective chapters, and the major findings are summarized below.

8.2.1 Effect of different heat treatments

The microstructures in the different heat-treated conditions consist of different combinations of α, β, α′ and α′′ phases, with various morphologies. The optimum tensile properties, along with lowest elastic modulus ~60 GPa, were observed for the sample solution treated at 900°C for one hour and water quenched. This decrease in elastic modulus is attributed to formation of α′ martensite along with retained β phase from fast cooling of the sample from β solution temperature. The elastic modulus value achieved in the present study is lowest among the various earlier reported heat-treated conditions of the alloy Ti-13Nb-13Zr. The elongation in the 900WQ condition was highest, and the elastic modulus was lowest (~60 GPa).

8.2.2 Surface modification via USSP technique

The process of USSP successfully developed nano size grains of ~21, ~13 and ~12 nm in the surface region of the alloy after 120, 240 and 360 seconds of USSP duration. TEM and XRD analysis confirmed the formation of nanostructure in the top surface region of the samples. No phase transformation was observed in the alloy after USSP treatment. Microhardness of the USSP treated specimen increased with increase in the USSP treatment duration and the depth of deformation also increased with increase in the USSP duration. With increase in the USSP duration, there is increase in the roughness and cracking tendency on the top surface of the samples. There is also an increase in compressive residual stress with increase in the USSP duration. However, residual stress is relieved from longer duration of USSP due to localized cracking.

8.2.3 Corrosion behaviour

It is important to point out that there is an increase in the corrosion resistance of all the USSP treated samples. However, decrease in passivation was observed after 30 s of USSP treatment, which can mainly be attributed to increased surface roughness and cracking tendency caused by the excessive plastic deformation. The best corrosion resistance was observed after 30 s of USSP treatment. XPS study of the USSP30 treated samples showed increase in the protective oxides (TiO_2, ZrO_2, and Nb_2O_5) formed on the surface compared to the Un-USSP condition. Electrochemical corrosion and static immersion tests both validated that 30 s of USSP duration improved the corrosion resistance significantly by forming an intact passive oxide layer and provided good corrosion resistance to the inner substrate layer. The grain refinement and surface roughness are the two opposing factors controlling the corrosion behaviour. Besides grain refinement, the presence of compressive residual stresses and the appropriate USSP duration produced optimal corrosion resistance. It may be concluded that by optimizing the compressive residual stress, grain size, and roughness at the surface of the alloy, the corrosion resistance can be improved significantly.

8.2.4 Low cycle fatigue behaviour

Cyclic hardening was observed after 100 cycles in the Un-USSP and USSP240 treated samples, whereas there was cyclic softening in the USSP240-SR samples. Following the USSP of 240 seconds, there was a threefold increase in the low cycle fatigue life, in particular at low strain amplitude (±0.55%) compared to that of the Un-USSP condition, due to overall positive effect of nano-structuring and the associated compressive residual stresses in the surface region, with little damage to the treated surface. Improvement in fatigue life was also observed in the USSP240-SR samples, though it was less (2.5 times) compared to the USSP240 samples due to reduced surface compressive residual stresses. Subsurface fatigue crack initiation was observed on fracture surface of the USSP treated fatigue specimens, unlike the Un-USSP and USSP240-SR samples in which crack initiation was observed from the surface. TEM micrograph showed that deformation of this alloy under cyclic loading occurs mainly through twinning, as revealed by large number of twins in the fatigue tested specimens.

8.2.5 Bio-compatibility

A significant increase in cell proliferation was observed with increase in the USSP duration. After 48 hours of incubation, there was about 1.6 times increase in cell viability of the MG-63 cells after 120 s USSP treatment. The cell proliferation was found maximum in the 120 s of USSP treated condition due to increased roughness and formation of nano-structures with high positive potential. Improvement in cell viability in all the USSP-SR samples can be attributed to the increased osteoblast anchorage due to combined effect of roughness and formation of biocompatible oxide layer on the surface via. stress-relieving treatment. A good amount of cell coverage can be observed in fluorescence microscopy images of the USSP and USSP-SR samples compared to the Un-USSP samples. USSP processing enhanced the roughness value in the Un-USSP sample, whereas USSP-SR treatment improved wettability and reduced contact angle, both of which are beneficial to cell survival and adhesion. This study showed that USSP treatment on alloy surface significantly changed the surface architecture to nano level, which enhanced the osteoblast cell adhesion and proliferation.

8.3 SUGGESTIONS FOR FUTURE WORK

Based on the present investigation, the following recommendations are made for future investigations:

1. Corrosion fatigue of samples in simulated body fluid.
2. Wear resistance of the Ti-13Nb-13Zr alloy in different heat treatment conditions.
3. Effect of different thermo-mechanical treatments on corrosion and fatigue behaviour of the Ti-13Nb-13Zr alloy.
4. High cycle fatigue behaviour of the Ti-13Nb-13Zr alloy in the Un-USSP, USSP and USSP-SR condition.