Development of Nickel Free Nitrogen Stabilized Austenitic Stainless Steel for Biomedical Applications



THESIS SUBMITTED IN PARTIAL FULFILLMENT FOR THE AWARD OF DEGREE

Doctor of Philosophy

by

Chandra Shekhar Kumar

DEPARTMENT OF METALLURGICAL ENGINEERING INDIAN INSTITUTE OF TECHNOLOGY (BANARAS HINDU UNIVERSITY) VARANASI-221005, INDIA

SUMMARY AND SUGGESTIONS FOR FUTURE WORK

7.1. Introduction

This chapter summarizes the important observations presented in the thesis and gives brief incitement of the scope of future research to be carried out.

7.2. Summary

Nickel causes allergy in humans and may impair the contacting cells. It is present in 316L austenitic stainless steel, which is widely used as temporary implant material. Nickel has potential to inhibit the growth of cells. Therefore, there is a need for the development of a nickel-free grade of austenitic stainless steel. The major findings are listed below:

- 1. Fe-Cr-Mn-Mo-N system (HNS-Mo), comprising carbon up to 0.05 wt %; Mn 19-20 wt %; Cr 19-20 wt %; Mo 0.50-1.0 wt%; N 0.60-0.70 wt%; Ni up to 0.10 wt%; Si up to 0.50 wt%; Cu up to 0.10 wt% and balance iron, was found well suited for biomedical applications than that of Fe-Cr-Mn-N system. HNS-Mo exhibits pitting potential comparable to that of 316L and shows higher strength and fatigue resistance than that of HNS and 316L, with acceptable *in vitro* and *in vivo* biocompatibility. This demonstrates the scope of development of austenitic stainless steel free from toxic elements and with improved properties, to be used as bioimplant material.
- 2. The higher pitting potential of the HNS-Mo as compared to that of the HNS is due to synergistic effect of nitrogen and molybdenum. Nitrogen causes higher lattice distortion in FCC lattice than that of carbon and there is short range ordering of Cr-N. Therefore, there is significant increase in strength of nitrogen containing HNS-Mo and HNS as compared to that of 316L. However, Mo present in HNS-Mo forms short range order with nitrogen which further enhances the strength and fatigue resistance.

- Overall, HNS-Mo austenitic stainless steel, with negligible nickel, was found to be a potent replacement of 316L, being used presently.
- 3. The effect of ultrasonic shot peening (USP) on the microstructure of the HNS was studied using XRD, SEM and TEM. The coarse grain microstructure of $36 \pm 6 \mu m$ was refined to nanoscale regime, in the surface, following USP. There was a progressive change in the microstructure from multidirectional twins to single direction twins, with depth from the USP treated surface, which ultimately disappeared after certain depth, depending on the shot diameter and the duration of USP. It is due to the decrease in strain and strain rate with increase in depth from the USP treated surface. High amount of manganese and nitrogen present in HNS, significantly decrease the M_{d30} temperature and restricts the deformation induced martensitic transformation and no phase transformation was observed even after 14 min of USP with 3 mm shots.
- 4. Effect of USP on corrosion resistance in SBF, *in vitro* cell culture and proliferation, hardness and low cycle fatigue behavior was studied. Corrosion resistance of the HNS in SBF was increased, following USP for shorter duration, whereas it was reduced from longer durations of USP due to excessive surface damage. For the HNS, 30 seconds and 1 minute of USP with shots of 3 mm and 2 mm diameters, respectively, are the optimum conditions for enhancement of corrosion resistance. HNS did not inhibit cell proliferation; rather, it improved up to some extent following USP. Hardness of the HNS was increased by 42% at the surface by 2 minutes of USP with 3 mm shots and was reduced gradually from surface towards interior.
- 5. There was a continuous decrease in LCF life of the USP treated samples of HNS with the duration of USP at the highest total strain amplitude ($\Delta\epsilon_t/2$) of \pm 0.80%; however, there was a marked increase in fatigue life at the lowest $\Delta\epsilon_t/2$ of \pm 0.40%. Fatigue life

- was increased by ~ 18 times at $\Delta \epsilon_t/2 = \pm 0.40$ % by the USP with 3 mm shots for 18 min duration. The marked enhancement in the LCF life at the lowest strain amplitude was mainly due to the delay in crack initiation from the nanostructured surface and the associated compressive residual stress.
- 6. The various surface properties of the HNS-Mo, like corrosion resistance in SBF, cell proliferation, high cycle fatigue and corrosion fatigue were assessed, following USP. Improvement in pitting corrosion resistance of the HNS-Mo was observed following USP. Nanostructured surface of the USP treated sample provides homogeneous elemental distribution that can promote rapid formation of Cr rich highly protective passive layer on the surface as compared to that of Un-USP sample. Also, a very large number of grain boundaries present on the surface leads to initial rapid dissolution of elements which in turn produces high amount of NH₄⁺ ions that promote passivation and inhibit the growth of pits. However, a longer duration of USP decreased the corrosion resistance as compared to the shorter duration. The best result was obtained for the USP 3-0.5 condition. The coverage of MG-63 cells on USP treated samples was higher than that of the Un-USP sample. Significant improvement in cell proliferation was observed on the USP 3-1 and USP 3-2 samples compared to the Un-USP, after 5 days of incubation for the MG-63 cells.
- 7. Endurance limit of the HNS-Mo was increased from 513 MPa to 572 MPa in air and from 475 MPa to 572 MPa in simulated body fluid environment, following 3 minutes of USP with 3 mm shots. Therefore, the negative effect of SBF on high cycle fatigue life of the HNS-Mo can be eliminated by USP.

7.3. Suggestions for Future Work

The following suggestions are made for future investigations, based on the present investigation:

- 1. HNS showed lower pitting potential as compared to that of 316L besides having plenty of nitrogen. The in-depth study can be performed for mechanistic explanation of this.
- The pH may change in the vicinity of implants during service due to infections and diseases. Therefore, the effect of pH on corrosion behavior of the HNS-Mo in an SBF environment can be studied.
- 3. The body fluids also contain protein which may significantly affect the corrosion behavior of stainless steel. Therefore, the effect of protein addition into the simulated body fluid on corrosion and corrosion fatigue behaviour of HNS-Mo can be studied.
- 4. Tribological behaviour of HNS-Mo in SBF environment may be studied.
- 5. Fretting fatigue of HNS-Mo in air and SBF environment may be studied.
- 6. Effect of magnetic field intensity on magnetic property of HNS-Mo may be evaluated.
- 7. Effect of USP on wear and fretting fatigue of HNS-Mo may be investigated.