CHAPTER – 9

Conclusions and Future Scope

9.1 Conclusions

A thorough study was investigated on the development of refractory element-based CCAs, focusing on their alloy design, microstructural characterization, mechanical properties, corrosion and wear behaviour, as well as in-vitro biocompatibility. The salient conclusions obtained from the study are as follows.

1. A new low density $Ti_{10}Nb_{30}Mo_{20}Cr_{20}Fe_{20}$ CCA was developed that exhibited a predominant BCC solid solution phase along with a minor amount of the C14-type Laves phase in its as-cast state. Upon annealing, the volume fraction of the Laves phase increased, accompanied by a microstructural transformation from a dendritic to a more or less globular morphology. In the as-cast condition, the dendritic regions exhibited higher hardness and elastic modulus compared to the interdendritic regions, owing to the segregation of harder and stiffer elements within the dendritic region. After annealing, the hardness and elastic modulus of the alloy were enhanced, primarily due to the higher amount of the hard Laves phase. Additionally, the corrosion resistance of the annealed CCA was improved, which may be attributed to greater microstructural homogeneity.
2. A novel $Ti_{35}Zr_{35}Nb_{15}Mo_5Fe_5Cr_5$ CCA was designed and developed to address the problem of high elastic modulus in the previous alloy. The microstructure of this alloy consisted of two BCC solid solution phases along with a C14-type Laves phase. This CCA demonstrated a superior combination of high strength and hardness without compromising plasticity, combined with a relatively low elastic modulus (97 GPa in the as-cast state). It also exhibited notably good wear resistance in simulated body fluid solutions. After annealing, strength, hardness, and wear resistance were further

enhanced. In addition, the dominant wear mechanism changed from delamination in as-cast condition to abrasive and oxidative wear after annealing at 1100°C.

3. The corrosion resistance of the as-cast and annealed $Ti_{35}Zr_{35}Nb_{15}Mo_5Fe_5Cr_5$ CCA was superior compared to that of conventional implant biomaterials such as 316L SS and cp-Ti. Additionally, the in-vitro biocompatibility assessments, including MTT assay and confocal microscopy imaging of MG-63 cells using various stains (AO/EtBr, DAPI, Rh-123, and DCFH-DA), showed that both the as-cast and 1100°C annealed CCAs exhibited enhanced cell adhesion and proliferation, and high cell viability compared to 316L SS and cp-Ti.
4. Crystallographic texture measurements were performed to unravel the underlying mechanisms and crystallographic orientation dependence of biocompatibility behaviour. It was found that texture plays a crucial role in influencing cell adhesion on the surface of the $Ti_{35}Zr_{35}Nb_{15}Mo_5Fe_5Cr_5$ CCA. As a result, the CCA annealed at 1100°C demonstrated higher cell adhesion and proliferation compared to the as-cast condition. This enhancement is attributed to the fact that, in the annealed alloy, a greater number of grains were oriented with their <111> direction normal to the surface exposed to cells (which was in turn related to the evolution of texture components such as γ-fiber). In contrast, the as-cast alloy contained relatively few grains with this favorable orientation. Since the <111> direction is a close-packed direction, it facilitates protein adsorption, thereby promoting cell adhesion in the annealed specimen enriched with <111>-oriented grains.
5. High-pressure torsion (HPT) deformation significantly increased dislocation density and lattice strain while reducing crystallite size. Additionally, the hardness of the CCA increased both from the center to the edge of the disc and with the number of

HPT turns. In contrast, the elastic modulus decreased considerably, from 97 GPa in the as-cast state to 68 GPa after 7 HPT turns. HPT deformation also improved corrosion resistance and in-vitro biocompatibility.

The outcomes of this entire study are schematically summarized in Figure 9.1, highlighting our research objectives and key achievements related to the development of novel CCAs for biomedical applications. As illustrated in Figure 9.1 (a), the primary goal was to develop a novel low-density CCA exhibiting high strength, low elastic modulus, excellent corrosion and wear resistance, and good biocompatibility, with a focus on potential biomedical applications.

Figure 9.1 (b) depicts the properties achieved with our initial composition, $Ti_{10}Nb_{30}Mo_{20}Fe_{20}Cr_{20}$ CCA, including low density, high strength, and good corrosion resistance. However, the elastic modulus of the alloy was quite high for biomedical applications, indicating that our goals were partially achieved. Therefore, it was concluded that the composition of the alloy needs further tuning. Subsequently, a new $Ti_{35}Zr_{35}Nb_{15}Mo_5Fe_5Cr_5$ CCA was developed, which demonstrated a superior combination of properties, including low density and elastic modulus, high strength with adequate plasticity, excellent corrosion and wear resistance, and superior biocompatibility, as shown in Figure 9.1(c).

Subsequently, despite achieving a comprehensive combination of properties, there was scope to further reduce the elastic modulus of the $Ti_{35}Zr_{35}Nb_{15}Mo_5Fe_5Cr_5$ CCA. To achieve this, high-pressure torsion (HPT) deformation was performed. As shown in Figure 9.1(d), HPT significantly reduced the elastic modulus while simultaneously enhancing the alloy's strength, corrosion resistance, and biocompatibility. Thus, the work embodied in this

thesis resulted in the development of a novel complex concentrated alloy with significant potential for biomedical applications.

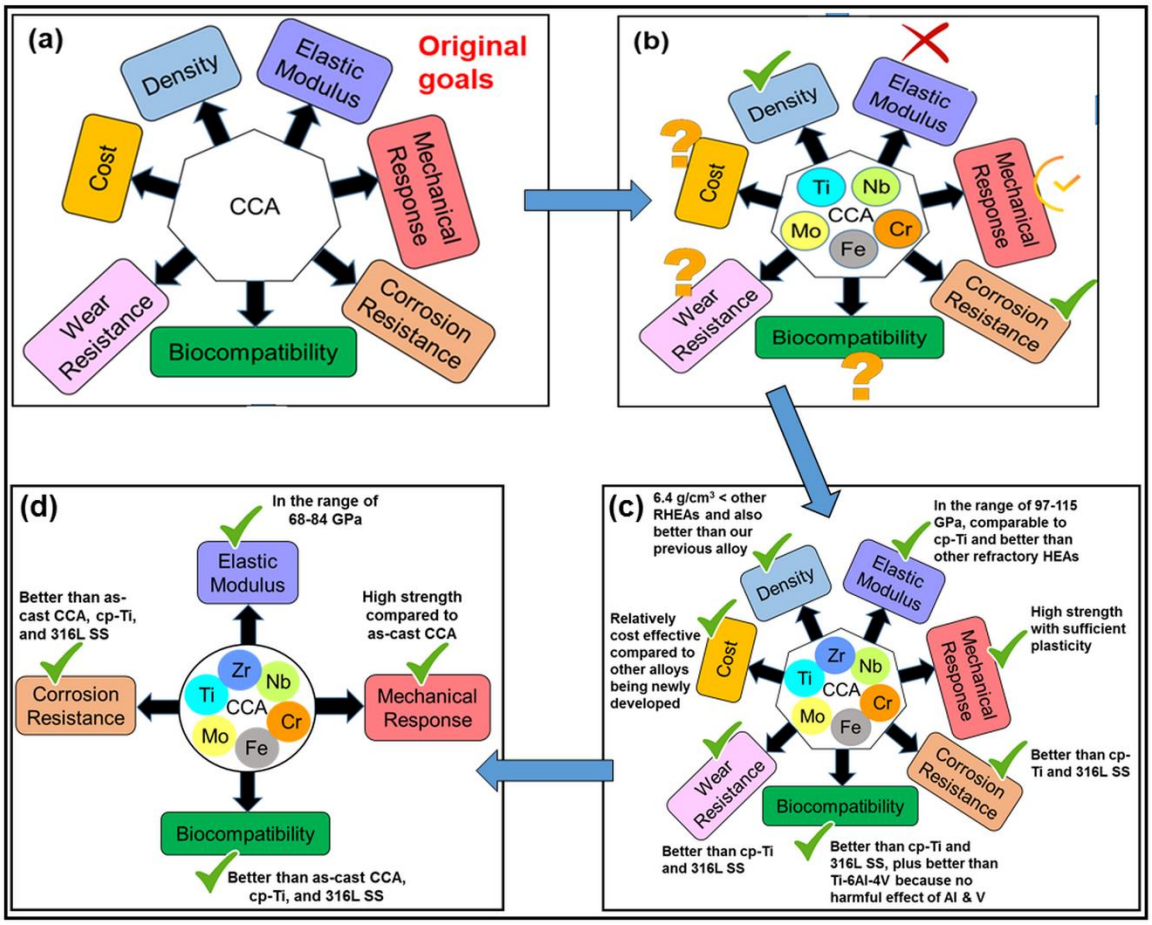

Figure 9.1 Schematic summarizing our research achievements with respect to the development of novel CCAs for biomedical application (a) initial targeted properties, (b) partially achieved in chapter 4, and (c and d) comprehensive property combination achieved in remaining result chapters.

9.2 Future Scope

Some of the limitations or challenges faced in the current work that may be improved upon in future, or aspects related to the work in this thesis that could be addressed in greater detail in the future are briefly summarized below.

1. In the present CCA, Cr was primarily added to enhance the corrosion resistance of the alloy; however, it is not considered fully biocompatible. Therefore, future alloy design efforts should focus on reducing or even suppressing Cr content while maintaining corrosion resistance through the incorporation of alternative alloying elements.
2. The developed CCA buttons fabricated by vacuum arc melting were small in size, due to which tensile specimens could not be obtained. Therefore, larger samples may be prepared in future using a furnace of suitable capacity to enable more rigorous and detailed mechanical testing including tensile and fatigue testing.
3. The elastic modulus of the CCA can be measured using ultrasonic techniques and further validated by evaluating this property through indentation methods.
4. Tribo-corrosion behaviour of CCA specimens in simulated body fluid solution may be performed.
5. Compare the properties of the present CCA with recently developed biocompatible alloys with Ti–Nb and Ti–Nb–Zr systems.
6. Biocompatibility testing in the current work was limited to in-vitro studies. Therefore, in-vivo biocompatibility testing may be conducted to assess the material's performance in biological environments in greater detail.

7. Future studies can focus on reducing the elastic modulus of the developed alloys by fabricating porous structures or architected lattices using additive manufacturing. Such approaches are particularly relevant for biomedical applications; as hip prostheses are increasingly being produced via additive manufacturing to achieve elastic modulus compatible with bone.