Phase Evolution, Thermal Stability, Mechanical, Wear and Biocompatibility Properties of High Entropy Steels and Fe-based High Entropy Alloys

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By

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This chapter summarizes the significant findings of the present work. The alloying behaviour, and thermal stability of the high entropy steel and Fe-based HEAs were critically analyzed. The detailed study on structural, microstructural, mechanical properties and surface properties of the SPSed samples of the high entropy steel and Fe-based HEAs were done. The key findings of the individual chapters were discussed at the end of the respective chapters.

7.1 Summary

The common summary of the chapter 3, 4 and 5 are described in the following section, $Fe_{40}Mn_{19}Ni_{15}Al_{15}Si_{10}C_1$, $Fe_{40}Mn_{14}Ni_{10}Cr_{10}Al_{15}Si_{10}C_1$, and $Fe_{40}Mn_{14}Ni_{10}Ti_{10}Al_{15}Si_{10}C_1$ high entropy steels were prepared successfully by mechanical alloying (MA) and further densified by spark plasma sintering. The phases formed after mechanical alloying are dual-phase mainly consisting of BCC and γ-brass type structure. In $Fe_{40}Mn_{19}Ni_{15}Al_{15}Si_{10}C_1$ this it also has B2-type phase after milling. HES milled powder sample showed the thermal stability up to 400 °C. This alloy has two exothermic events at 420 °C and 520 °C, which is correlated with ordering and/or texting of γ-brass type structure and the formation of Fe_5Si_3–type silicides, respectively. The additions of Cr (HES2), resulted in thermal stability up to 400 °C. However, at higher temperatures it forms the FCC solid solution, Cr_3Si, and $Cr_{23}C_6$. The addition of Ti (HES3), ensued thermal stability up to 500 °C. However, at elevated temperatures, it leads to the formation of FCC solid solution, TiC and Fe_5Si_3.

The phases formed in the SPSed sample of HES1 consisted of dual-phase structure containing BCC and B2-type along with Fe_5Si_3. The mechanical properties like microhardness, compressive strength and strain of the alloy were evaluated and it was found to be 7.8 GPa, 2000 MPa and 19 %, respectively. By adding Cr, it leads to the formation of FCC and B2-type with Cr_3Si and $Cr_{23}C_6$ as precipitates. The addition of Cr leads to decrement in the strain, and its value was found to be 7

% may due to the formation of hard intermetallics like Cr_3Si and $Cr_{23}C_6$. The mechanical properties like microhardness and compressive strength were increased by 33 % and 16 % as compared with HES1. The increase in mechanical properties (HES3) is due to the formation of dual-phase structure consisting of FCC and B2 coexisted with TiC and Fe_5Si_3 nanoprecipitates after SPS, simultaneously the slight decrease in ductility was observed. The dominant strengthening mechanism is grain boundary and dislocation in this high entropy steel. In HES2 and HES3, the precipitates strengthening is dominant strengthening mechanism. In all the three high entropy steels, the HES3 (containing Ti) has better mechanical properties with good wear resistant and biocompatibility. However, these alloy systems have been the potential candidates for high temperature structural and implant applications.

$Fe_{40}Mn_{20}Cr_{20-x}Ni_xTi_{10}Al_{10}$ (x = 0, 5, and 10 at. %) HEAs were successfully prepared using the mechanical alloying and subsequently by spark plasma sintering. The systematic investigations have been done to understand the effect of Ni and Cr on the structure, microstructure, mechanical, wear and biocompatibility of the non-equiatomic Fe-based HEAs. The alloying behaviour during milling was established through XRD. It was observed that the dual-phase of BCC and χ-phase type structure was formed during the milling in all the three alloy systems. It was observed from ex-situ XRD of the annealed sample at 600 °C that the FCC solid solution was evolved in HEA1, HEA2, and HEA3. Lattice parameters of BCC and FCC phase slightly decreased as Ni content was increased in the non-equiatomic HEAs. After SPS, the phase transformed from BCC to FCC as the Ni content increased from x = 0 to 10 at. %, simultaneously the yield strength decreased but ductility increased. As the microhardness of the SPSed samples of the non-equiatomic HEAs decreased, the specific wear rate increased. HEA2 showed better biocompatibility in MG-63 cell line as compared with other alloy systems. Among the three alloys systems, HEA2 is the best potential candidate for biomaterial applications, as this alloy exhibited the good combination of strength and ductility with good wear resistance and biocompatibility. The comparative assessment of the various high entropy steels and Fe-based HEAs with 316L in terms of mechanical and wear properties of the SPSed samples. Table 7.1 mentioned the comparative assessment of the various present alloy systems, and 316L in terms of mechanical and wear properties of the SPSed systems.

Table 7.1: The comparative assessment in terms of hardness, elastic modulus, yield compressive strength, and wear rate of the various high entropy steels, Fe-based HEAs and 316L.

Alloys	**Microhardness (MPa)**	**Elastic modulus (GPa)**	**Y. S. (MPa)**	**Wear rate (mm^3/mN)**
HES1	7.8	184	2046	1.79×10^{-5}
HES2	7.4	189	1954	1.99×10^{-5}
HES3	**10.4**	209	2305	**1.62×10^{-5}**
HEA1	6.4	183	1962	1.30×10^{-4}
HEA2	**5.7**	**131**	**1810**	1.65×10^{-4}
HEA3	4.2	127	1300	4.21×10^{-4}
316L	2.4	210	501	0.4×10^{-4}

7.2 Suggestions for future works

- Careful optimization of milling parameters is required to dissolution of undissolved Si in BCC or gamma brass type structure.

- Due to the vast compositional space of high entropy steel and Fe-based HEAs, the alloy design and development should be optimized as per the desired requirements in terms of microstructure and properties using the machine learning technique.
- Detailed failure analysis of the fractured sample after the compression test can be carried out. This will help in understanding the failure mechanism in this alloy systems.
- The tensile test of the SPSed samples is to be carried out to advocate its applicability as a structural material.
- Further optimizations of the composition can be done to enhance the room temperature ductility without compromising strength.
- For biomaterial application, further analysis of the tribological and corrosion studies in the body fluid as well as leaching behaviour of the elements can be carried out.