

CHAPTER 8

CONCLUSION

This chapter outlines the conclusions derived from the present investigations on the synthesis of (Fe-Ni)-ZrO₂ Metal Matrix Composites via powder metallurgy route and auto-combustion followed by reduction method and thereupon mechanical & electrochemical characterizations as discussed in the chapters 4 to 7.

The conclusions on the formation of Fe_(100-x)Ni_(x) alloy (x= 10, 20, 30, 40 and 50 wt%) and mechanical and electrochemical characteristics as described in Chapter 4 are:

1. Fe_(100-x)Ni_(x) alloys with varying x from 10 to 50 wt% has successfully synthesized of using powder metallurgy route by sintering at 1000°C/1h, 1200°C/1h and 1250°C/1h.
2. Presence of α and γ -(Fe,Ni) phases are observed in XRD patterns of Fe_(100-x)Ni_(x) specimens. Similar observations of the presence of α and γ phases are also evident from microstructural analysis. Increase in γ -(Fe,Ni) is evident with increasing both Ni content and sintering temperature. Maximum γ -(Fe,Ni) formation is observed in Fe₅₀Ni₅₀ after sintering at 1250°C/1h.
3. The density of the alloy specimens decreases due to higher γ phase formation with increasing Ni content, however density increases with increasing sintering temperature due to the reduction in defect concentration and reduced porosity.

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4. Specimens containing more γ phase in compositions with higher Ni content show lower hardness, however hardness increases with increasing sintering temperature due to the reduced porosity and better cohesion of α and γ phases.
 5. Wear rate is influenced by the composition, sintering temperature, and the applied load. Wear rate increases with increasing applied load. Excess of α phase results in the elimination of hard craters from the specimen surface in $\text{Fe}_{90}\text{Ni}_{10}$ and $\text{Fe}_{80}\text{Ni}_{20}$ compositions. Higher Ni-containing specimens $\text{Fe}_{60}\text{Ni}_{40}$ and $\text{Fe}_{50}\text{Ni}_{50}$ show higher wear loss due to the presence of higher fraction of softer γ phase. Maximum wear resistance is observed in $\text{Fe}_{70}\text{Ni}_{30}$ due to the optimum content of both γ and α phases, which support each other during sliding wear.
 6. Formation of $\gamma\text{-(Fe,Ni)}$ resulted in the decrease in corrosion current of $\text{Fe}_{(100-x)}\text{Ni}_x$ alloy specimens, when examined in 3.5 wt% NaCl solution. Therefore, maximum corrosion resistance is observed in alloy composition $\text{Fe}_{50}\text{Ni}_{50}$ due to maximum $\gamma\text{-(Fe,Ni)}$ phase formation. The corrosion resistance for the alloy composition $\text{Fe}_{70}\text{Ni}_{30}$ was also good and comparable to $\text{Fe}_{50}\text{Ni}_{50}$.

The conclusions on the formation of $(\text{Fe}_{70}\text{Ni}_{30})\text{-ZrO}_2$ Metal Matrix Composites ($\text{ZrO}_2 = 0, 2.5, 5, 10$ and 15 wt %) and mechanical & electrochemical characteristics as described in chapter 5 are:

1. $(\text{Fe}_{70}\text{Ni}_{30})\text{-ZrO}_2$ composites with varying ZrO_2 content (0, 2.5, 5, 10 and 15 wt%) were successfully synthesized using powder metallurgy route and sintering at $1150^\circ\text{C}/3\text{h}$.

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2. XRD patterns show the presence of γ -(Fe,Ni), α -(Fe,Ni) and ZrO_2 peaks after sintering. Increase in ZrO_2 peaks is observed with increasing ZrO_2 content in the composition. No intermediate phase formation between metal and ZrO_2 particles is evident from XRD. From the SEM analysis, ZrO_2 particles are observed along the grain boundary region and near the pores.
 3. The density of composites specimens decreases with the addition of ZrO_2 particles, which has a lower theoretical density as compared to Fe and Ni.
 4. Addition of ZrO_2 reinforcement helps in retarding the plastic deformation of $\text{Fe}_{70}\text{Ni}_{30}$ matrix. This increases the hardness of composites. Maximum hardness 119 Hv is observed in the composite containing 15 wt% ZrO_2 reinforcement.
 5. Presence of ZrO_2 particles in $\text{Fe}_{70}\text{Ni}_{30}$ matrix increases the wear resistance of composites. The increase in ZrO_2 content up to 10 wt% enhanced the wear resistance of composites. The addition of more ZrO_2 content, i.e., 15 wt% results in a decreased wear resistance.
 6. In (Fe-Ni)- ZrO_2 composites, the segregation of ZrO_2 particles at pores and grain boundaries as evident from SEM micrographs possibly retarded the attack of the electrolyte solution. Therefore, the increase in ZrO_2 concentration up to 10 wt% results in improved corrosion resistance of composites. For 15 wt% ZrO_2 containing composites, the increase in I_{corr} results in reduced corrosion resistance due to the weak metal-ceramic interface at higher reinforcement content.

The conclusions on the formation of $\text{Fe}_{(100-x)}\text{Ni}_{(x)}$ alloy powders (x= 10, 30 and 50 mole%) using sol-gel auto-combustion route followed by reduction as described in Chapter 6 are:

1. $\text{Fe}_{(100-x)}\text{Ni}_{(x)}$ nanocrystalline alloy powders (x= 10, 30 and 50 mole%) were successfully synthesized using a wet chemical route (sol-gel auto-combustion) followed by hydrogen reduction.
2. Formation of α and γ -(Fe,Ni) phases were observed from XRD analysis. Complete γ -(Fe,Ni) formation is achieved in $\text{Fe}_{50}\text{Ni}_{50}$ alloy powder. The increase in saturation magnetization (Ms) supports the formation of γ -(Fe,Ni) phase with increasing Ni content. Maximum Ms is observed in $\text{Fe}_{50}\text{Ni}_{50}$ alloy powder. Presence of particles below 50 nm in alloy powders is confirmed from TEM results.

The conclusions on the formation of $(\text{Fe}_{70}\text{Ni}_{30})\text{-ZrO}_2$ composites by the addition of nano ZrO_2 ($\text{ZrO}_2= 0, 2.5, 5, 10$ and 15 wt%) particles in chemically prepared nano-crystalline $(\text{Fe}_{70}\text{Ni}_{30})$ alloy powder as described in chapter 7 are:

1. A- $(\text{Fe}_{70}\text{Ni}_{30})\text{-ZrO}_2$ composites were successfully synthesized via powder metallurgy route using ZrO_2 nano powder (0, 2.5, 5, 10 and 15 wt%) and chemically prepared $\text{Fe}_{70}\text{Ni}_{30}$ alloy nano powder and sintering at $900^\circ\text{C}/1\text{h}$.
2. XRD patterns show the presence of γ -(Fe,Ni), α -(Fe,Ni) and ZrO_2 peaks in sintered composites. Increase in ZrO_2 peaks are observed with increasing ZrO_2 content in the composition. No intermediate phase formation between metal and ZrO_2 particles is evident from XRD.

TABLE 8.1: Comparative results of prepared specimens

S.No.	Specimen	Density (gm/cm ³)	Hardness (Hv)	Corrosion rate (mpy)
1	Fe ₇₀ Ni ₃₀	6.34	91	41.6
2	(Fe ₇₀ Ni ₃₀)-10ZrO ₂	5.65	111	28.4
3	A-(Fe ₇₀ Ni ₃₀)	6.77	118	32.4
4	A-(Fe ₇₀ Ni ₃₀)-15ZrO ₂	6.45	158	8.3

3. Segregation of nano ZrO₂ particles is observed at the pore and boundary region in the micrographs.
4. Maximum density is achieved in A-(Fe₇₀Ni₃₀) sintered specimen. Addition of ZrO₂ particles resulted in a slight decrease in density of composite.
5. The addition of nano ZrO₂ particles in chemically prepared Fe₇₀Ni₃₀ matrix improved the hardness of composites. Maximum hardness 158 Hv is observed in composite containing 15 wt% ZrO₂ content.
6. For the composites A-(Fe₇₀Ni₃₀)-ZrO₂, presence of nanoparticles at grain boundaries and pores results in enhanced corrosion resistance. The maximum corrosion resistance is evident in A-(Fe₇₀Ni₃₀)-15ZrO₂.

It is concluded that the addition of inert phase (ZrO₂ particles) in Fe-Ni alloy matrix helps in improving the hardness, wear, and corrosion characteristics. When micro ZrO₂ particles are used for reinforcement, the improvement in wear and corrosion characteristics is observed for the composites up to 10 wt% reinforcement content. For 15 wt% reinforced composites, the decrease in properties is observed. The use of nanoparticles as both matrix and reinforcement phases results in better properties than the micron size particles reinforced composites. In this case, all the features are observed improving up

to 15 wt% ZrO₂ addition. The relative improvement in properties can be concluded from Table. 8.1. It is expected that the present investigation will be helpful in developing metal matrix composites for heavy duty and tribological applications.