Chapter 10

Conclusions and Scope for Further Research Work

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The chapter outlines the salient conclusions derived from the present research work on Fe-Al_2O_3 Metal Matrix Nanocomposites, presented in the preceding chapter 5 to 9. It also attempts to identify scope for further research work in the area of Iron based Metal Matrix Nanocomposites.

10.1 *Conclusions*

The aim of investigations was to explore the possibility of developing least explored Al_2O_3 reinforced Fe matrix composites for heavy duty applications. In the present investigations, it was initially aimed to synthesize Fe-Al_2O_3 metal matrix composites with varying concentration of alumina (5-30 wt%) by powder metallurgy route and to characterize them for their structural and mechanical behavior. From these investigations, we could find optimum compositions and processing parameters for Fe – Al_2O_3 Metal Matrix Nanocomposites, suitable for further investigations from applications point of view. The next stage of investigations was aimed to study the effect of doping (CoO and CeO_2) on the properties of one of these selected compositions (Fe-10% Al_2O_3) of nanocomposites. Apart from this, in order to study the effect of addition of secondary ceramic reinforcement, Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite specimens were also synthesized by powder metallurgy technique. Finally, the environmental degradation of the above nanocomposites was investigated by studying their corrosion behavior. In these investigations, it was aimed to study the effect of processing parameters on the corrosion behavior of selected composition (Fe-5/10% Al_2O_3) of metal matrix nanocomposites. Corrosion behavior of (Fe-10% Al_2O_3) composite was also selected to study the effect of doping (CoO and CeO_2) on their corrosion characteristics.

The detailed conclusions deduced from the investigations carried out in the present thesis are described under the following four categories:

Physical Behavior

- Values of density of Fe-Al_2O_3 nanocomposites revealed that the densities were found to improve upto 10 wt% of the Al_2O_3 reinforcement. Beyond 10 wt% of Al_2O_3 reinforcement i.e. for 20 and 30 wt% of Al_2O_3 reinforcement the density decreased. 20 wt% and 30 wt% Al_2O_3 reinforced specimen were found to be brittle in nature and thus showed low strength.

- Doping of CoO and CeO_2 in Fe-Al_2O_3 metal matrix nanocomposites gives better densification.

- Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite specimens show improved density values in comparison to Fe-Al_2O_3 and CoO doped Fe-Al_2O_3 nanocomposite system.

From the investigations on the density of different iron-alumina nanocomposite systems in the present thesis it was found that the density of the formed nanocomposites depends upon the composition and processing parameters i.e. sintering temperature and time. Density of the Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite specimens was highest.

Structural Behavior

- In Fe-Al_2O_3 nanocomposite system, XRD results show the presence of iron aluminate ($FeAl_2O_4$) phase. This iron aluminate phase is formed as a result of reactive sintering between iron and alumina particles.

- Amount of the iron aluminate phase increases with increase in the percentage of alumina reinforcements. The amount of iron aluminate phase was found to be high in the specimen with 30 wt% of Al_2O_3 reinforcement.

- The amount of the iron aluminate phase also depends upon the sintering temperature and time respectively. On increasing the sintering temperature, the amount of the iron aluminate phase increases.

- Results of SEM confirm findings of the XRD. Scanning Electron Microscopic studies of Fe-Al_2O_3 composite reveal the formation of highly dense phase. Further, the high magnified view of the same micrograph shows the presence of nano iron aluminate phase. The amount of this iron aluminate phase increased with the content of alumina and sintering temperature.

- In CoO doped Fe-Al_2O_3 nanocomposite system, XRD results show that on increasing the percentage of CoO from 0.5% to 1.0% the amount of iron aluminate ($FeAl_2O_4$) phase decreased significantly.

- SEM micrographs of CoO doped Fe-Al_2O_3 nanocomposite system shows the presence of constituent phases such as Fe and Al_2O_3. CoO in the present case has been found to be trapped in between the Fe and Al_2O_3 particles. Due to this entrapment rate of reaction between these two decreases due to which formation of nano iron aluminate phase has reduced significantly.

- In CeO_2 doped Fe-Al_2O_3 nanocomposite system, XRD results show that on increasing the percentage of CeO_2 from 0.5% to 1.0% the amount of iron aluminate ($FeAl_2O_4$) phase increases with the presence of separate peaks of CeO_2.

- SEM of CeO_2 doped Fe-Al_2O_3 nanocomposite system showed the presence of particles of cerium oxide (confirmed by EDAX) in the minute pores of the specimen. Dense phase structure was found for 0.5 and 1.0 wt% of CeO_2 doped nanocomposite specimens. Energy dispersive spectroscopy studies reveal the presence of all the elements in the specimens.

- In Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite system, iron aluminate ($FeAl_2O_4$) and iron zirconium oxide (Zr_6Fe_3O) phases were found. SEM results also show the nano size particles of constituent phases. At low sintering temperature, a small amount of iron aluminate and iron zirconate phase forms irrespective of time of sintering.

Investigations on the structural behavior of different iron-alumina nanocomposite systems reveal that constituent and reactive phases are present in the specimens. Sintering in the present case is found to be reactive. Reactivity depends upon the processing parameters i.e. sintering temperature and time as well. It also depends upon the type of doping. In doped and undoped Fe-Al_2O_3 composite systems, iron aluminate ($FeAl_2O_4$) phase forms whereas in hybrid Fe-Al_2O_3-ZrO_2 nanocomposite system iron aluminate and iron zirconate phases form due to reactive sintering. Electron micrographs of various specimens showed dense phase composite structure with the dispersion of nano reactive phases.

Mechanical Behavior

- Hardness of the Fe-Al_2O_3 nanocomposite specimens was found to be dependent upon the formation of iron aluminate phase. Amount of iron aluminate phase in turn depends upon sintering temperature and time.

- Hardness numbers of CoO doped Fe-Al_2O_3 nanocomposite specimens were found to be comparable to that of un-doped Fe-Al_2O_3 nanocomposite specimen. Hardness of CoO doped Fe-Al_2O_3 nanocomposite specimen was found to decrease on increasing the percentage of CoO from 0.5% to 1.0%.

- CeO_2 doped Fe-Al_2O_3 nanocomposite specimens showed the highest hardness value. Hardness value increased on increasing the percentage of cerium oxide from 0.5% to 1.0%.

- Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite specimens showed improved hardness number in comparison to Fe- Al_2O_3 and CoO doped Fe- Al_2O_3 metal matrix nanocomposite specimens.

- Wear rate of the Fe-Al_2O_3 nanocomposite specimen under dry sliding wear test was found to increase with increase in the load on the test rig. For 5% Al_2O_3 reinforced specimen at low load adhesive wear was more prominent whereas at higher load abrasive wear was more active. For 10% Al_2O_3 reinforced nanocomposite specimen, removal of the material is due to fragmentation of asperities and also due to cutting and flowing actions of penetrated hard asperities into the softer surface.

- For the nanocomposite specimen having 0.5% CoO an adhesive wear was found upto a load of 1.5 kg but at 2.0 kg load the wear behavior was abrasive in nature. The specimen with 1.0% cobalt oxide showed the abrasive wear under all loads. This may be due to the reason that the bonding between different particles in the nanocomposite specimen is small and particles come in between the specimen and the disc causing abrasive wear under lower loads.

- For CeO_2 doped nanocomposite specimen, values of wear rate were found to reduce with the increase in the percentage of cerium oxide. The removal of the material was found due to the tangential skiving of the iron aluminate phase on the specimen surface. The removed debris material generates peel off marks on the specimen surface.

- In Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposite system, values of wear rate were found to improve. SEM images of the worn surfaces showed the light scoring marks on the specimen surface. Intensity of the marks was extremely low as compared to Fe-Al_2O_3 and CoO doped Fe-Al_2O_3 metal matrix nanocomposites.

- Studies on deformation behavior of Fe-5% Al_2O_3 nanocomposite specimens were carried out by varying the h/d ratio under different interfacial frictional conditions. It was revealed that the dry specimens show more bulging than the lubricated ones. The test specimen show more surface movement on top surface than the bottom surface under all the deforming conditions. A significant improvement in the density and hardness is observed in all the deformed test specimens.

Discussions on mechanical behavior of different iron-alumina nanocomposite systems reveal that hardness, wear and deformation of the system depend on the composition, processing parameters and dopant along with its concentration. Hardness increases with an increase in the addition of Al_2O_3 upto 10 wt.%, thereafter it decreases. Wear mechanism maps reveal different types of behavior i.e. adhesive, abrasive, microploughing, microfracturing etc. Wear mechanism depends on the composite system and the load during wear. Deformation behavior of the specimens varies with the height to diameter ratio of the specimen. It also depends upon the different interfacial frictional conditions.

Electrochemical Behavior

- Corrosion behavior of 5% Al_2O_3 reinforced specimen showed that due to Al_2O_3 reinforcement, the Tafel plots shift towards lower current regions, thereby, denoting the improvement in the corrosion resistance of the specimens. XRD and SEM analysis revealed the formation of the aluminum chlorate ($AlCl_3O_{12}$) phase which is formed as a result of the attack of HCl on aluminum oxide particles. Anti-corrosion efficiency of all the specimens was found to be above 90%.

- Corrosion behavior of 10% Al_2O_3 reinforced specimen showed that iron aluminate ($FeAl_2O_4$) phase formation improved the corrosion resistance of Fe-Al_2O_3 metal matrix nanocomposite specimens. XRD results showed the presence of aluminum chlorate and iron chloride phases respectively in the corroded specimens. SEM results showed that the specimen having poor corrosion characteristics showed the formation of dark patches on the specimen surface.

- Corrosion behavior of CoO doped Fe-Al_2O_3 nanocomposite specimen showed that CoO doping improves the corrosion resistance of pure Fe as well as of Fe-Al_2O_3 metal matrix nanocomposite specimens. XRD results show the presence of aluminum chlorate and cobalt chlorate phases respectively in the corroded specimens. Presence of nano particles were found in the composite specimens with high percent of cobalt oxide and nano rods in the pure Fe specimen with 0.5% of cobalt oxide. Anti-corrosion efficiency of 1.0% doped nanocomposite specimen and 0.5% doped pure iron specimen was found to be 99.99%.

- Corrosion behavior of CeO_2 doped Fe-Al_2O_3 composite specimen shows that CeO_2 doping improves the corrosion resistance of pure Fe as well as of Fe-Al_2O_3 metal matrix nanocomposite specimens. Best corrosion resistance was found for the specimen having 0.5% of CeO_2 doping. Most of the specimens show the presence of amorphous phase. 0.5% CeO_2 doped nanocomposite showed the anti-corrosion efficiency of 99.99%.

Investigations on the electrochemical behavior of various iron-alumina metal matrix nanocomposites depend upon the percentage of reinforcement, type of dopant and concentration of the dopant. Anti-corrosion efficiency of nanocomposite specimens is higher in comparison to pure iron specimens prepared by powder metallurgy technique. Doping of CoO and CeO_2 in the iron-alumina nanocomposite with optimum concentration greatly increases anti-corrosion efficiency. This increase in anti-corrosion efficiency is reflected by shift in corrosion potential and formation of nano structured aluminium chlorate ($AlCl_3O_{12}$) protective layer on the specimen.

In summary the present thesis reports the successful synthesis of Fe-Al_2O_3, CoO and CeO_2 doped Fe-Al_2O_3 and Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposites via powder metallurgy technique.

Fe-Al_2O_3 metal matrix nanocomposite specimens synthesized by sintering in the temperature interval of 900 - 1100°C for 1 – 3 hours showed the presence of iron aluminate phase due to reactive sintering. Iron aluminate phase formation depends upon the sintering temperature and time respectively. High amount of iron aluminate as is present in (20/30%) Al_2O_3 reduces the bonding strength between the particles and thus increases the brittleness. 5% and 10% of Al_2O_3 reinforced metal matrix nanocomposite show better hardness, wear, deformation and corrosion characteristics respectively.

During the investigation on CoO and CeO_2 doped Fe-(10%) Al_2O_3 nanocomposites synthesized by sintering at 1100°C for 1 h, it was found that the doping of cobalt oxide reduced the iron aluminate phase formation and cerium oxide doping enhanced the iron aluminate phase formation. Mechanical and electrochemical properties of both CoO and CeO_2 doped specimens were found to improve. Anti-corrosion behavior of 1.0% doped CoO and 0.5% doped CeO_2 specimens was found to be highest among all the composite specimens.

Successful synthesis of Fe-Al_2O_3-ZrO_2 hybrid metal matrix nanocomposites was also done via powder metallurgy technique. Specimens were compacted and sintered in the temperature range of 900-1100°C for 1-3 hour respectively. Iron aluminate and iron zirconium oxide phases were formed due to the reactive sintering between the iron – alumina and iron – zirconia particles respectively. Hardness values were found to be high in comparison to Fe-Al_2O_3, Fe-ZrO_2 and CoO doped Fe-Al_2O_3 metal matrix nanocomposites. Wear rate of the specimens were found to be reduced in comparison to the synthesized Fe-Al_2O_3 metal matrix nanocomposites. Specimens showed improved mechanical properties in comparison to both iron-alumina and iron-zirconia metal matrix nanocomposites system.

It is expected that the outcome of the present investigation will be helpful in designing and developing metal matrix nanocomposites for heavy duty applications.

10.2 Scope for Future Work

On the basis of above investigations there is an ample amount of scope for further research work for the development of iron based metal matrix nanocomposites. Some of the scope for future work is listed below:

- Synthesis of a few more specimens can be done using different dopants such as titanium di-oxide etc. After the successful synthesis of these specimens, similar characterizations may be carried out.

- Wear behavior of the same specimens can be carried out by varying the sliding velocity and keeping the sliding time and load as a constant factor.

- Deformation behavior studies of the same specimens can be carried out by using some semi solid lubricants such as vaseline etc.

During the completion of Ph.D. thesis it was also found that other researchers have also taken up the current area to extend the investigations in other directions. Outcome of their extended work is listed as follows:

- U. J. Prasanna Kumar, M. Tech. Thesis (2014) entitled "Study of Closed Die Deformation Behaviour of Cylindrical Fe-Al_2O_3 Metal Matrix Composites (MMCs)", Department of Mechanical Engineering, Indian Institute of Technology (Banaras Hindu University), Varanasi-INDIA.

- Vinod Gupta, M. Tech. Thesis (2014) entitled "Plane Strain Deformation Studies of Fe-Al_2O_3 Metal Matrix Composite Rectangular Preforms Under Different Interfacial Frictional Conditions", Department of Mechanical Engineering, Indian Institute of Technology (Banaras Hindu University), Varanasi-INDIA.

- Khan and Siddiqui (2014) synthesized Al-Fe-Cr-Al_2O_3 composites through mechanical alloying with varying (10%-30%) weight percentages of aluminium oxide reinforcement citing our publication no. (03). Results obtained shows that 20 wt. % alumina yields the highest crystalline size refinement and also has the maximum lattice strain.