PREFACE

New and advanced materials refer to class of materials and modifications to existing materials to obtain superior performance. Materials structured at the nanometer scale have become an interesting area of research due to their unique and expected macroscopic characteristics. Composites, metal or ceramic matrix and intermetallics which fall under the category of advanced materials are receiving increasing attention due to their higher specific strengths, stiffness and high temperature properties. The interest in finding superior reinforcements for metallic matrices has been growing considerably over the past few years, largely focusing on investigating their contribution to the enhancement of the mechanical properties of metal matrix composites. On the other hand, intermetallic compounds which are emerged as materials with vast potential for application in wide range of technologically important areas but they are associated with the reduced dislocation activity resulting in low ambient temperature ductility and fracture toughness, which restricts large-scale industrial or structural applications of these intermetallics. Hence, several attempts have been made in recent years to alleviate this problem, and this still remains a major challenge for the material scientists to overcome this hurdle. The ways to improve the room temperature ductility of intermetallics includes reduction in grain size, disordering of the lattice, improving the dislocation motion and modifying the crystal structure of the phase into a more symmetric.

Non-equilibrium processing of materials is found to be a novel technique to synthesize materials and/or devices with new properties and control of the structure of the materials in order to provide a precisely tailored set of properties. The physical and mechanical properties of the materials synthesized by this route are known to have improved properties in comparison with the conventional ingot processed materials. Those materials which are conventionally quite strong but very brittle, such as intermetallics and ceramics can have enhanced ductility from grain size reduction. Therefore, one can design the alloys by controlling the microstructure in the nanoscale. Considering these points, the objective of this work is to study the structural changes occurring in aluminum based composites and intermetallics during milling and to analyze the strengthening effect.

The present thesis is divided in seven chapters. **Chapter-1** presents a brief introduction along with the review of aluminum based alloys, their properties and applications. This chapter gives the detail about the aluminum matrix composites, their processing routes and strengthening mechanisms and also on iron aluminide intermetallics. Further the chapter demonstrates the basic understanding about mechanical alloying/milling (MA/MM) and production of nanostructured and amorphous materials by this process. Mechanical properties of nanostructured materials have been discussed in details in the present chapter. It also includes a basic idea about deformation mechanisms involved in nanostructured materials to understand the inverse Hall-Petch (IHP) effect an attribute observed in materials at mesoscopic scale.

Chapter-2 deals with the detailed experimental procedure followed for the preparation and characterization in the present research work. Aluminum based crystalline, composite alloys and intermetallic compounds are of special interest in the present experimental work. Aluminum based composites were synthesized by mechanical alloying and aluminum based intermetallics were synthesized by arc melting techniques in the present work. The prealloyed iron aluminide intermetallics were further processed by non-equilibrium processing such as mechanical milling using planetary ball mill. The characterization of as-cast alloys and MA/MM powder samples were done using various techniques. Among the characterization techniques phase identification, crystallite size and sequence of phase evolution at different stages of MA/MM was done by X-ray diffraction (XRD) techniques with an automated D8 Discover Bruker diffractometer using CuK_{α} radiation (λ = 0.15406 nm) operated at 40kV/30mA. The microstructures were characterized using an scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDX), transmission electron microscope (TEM) and high resolution transmission electron microscope (HRTEM). The thermal stability of the samples was investigated by differential thermal analysis (DTA). Microhardness was measured using standard Vickers microhardness tester and nanoindentaion using a MTS Nano Indenter XP.

Chapter-3 presents the investigation of morphology, structure and hardness variations of garnet reinforced EN AW6082 aluminum alloy composites. High-energy ball milling of EN AW6082 Al-alloy powder, with and without garnet reinforcement, was performed under argon atmosphere for various durations up to 50 hour. The study aimed at exploring the role of alloying elements and hard reinforcement particles on the structural evolution at different stages of mechanical milling. The composite powders were characterized in terms of the morphological variation, microstructural evolution, and thermal stability. Microindentation and nanoindentation measurements were carried out on the individual powders as well as composite particles to estimate the changes in the mechanical properties of the composites with milling time. The results reveal that incorporation of hard garnet particles hastens the milling effect and leads to significant improvement in hardness and modulus of unreinforced pure aluminum and aluminum alloy.

Chapter-4 presents the influence of milling time on the structure, morphology and thermal stability of multi-walled carbon nanotubes (MWCNTs) reinforced EN AW6082 aluminum alloy powder. Structural and microstructural characterization of the mechanically milled powders and micro- and nano-hardness of the composite powder particles were evaluated. The morphological and x-ray diffraction studies on the milled powders revealed that the carbon nanotubes (CNTs) were uniformly distributed and embedded within the aluminum matrix. No reaction products were detected even after long milling up to 50 h. Nanotubes became shorter in length as they fractured under the impact and shearing action during the milling process. A high hardness of about 436 ± 52 HV is achieved for the milled powders, due to the addition of MWCNTs, after milling for 50 h. The increased elastic modulus and nanohardness can be attributed to the finer grain size evolved during high energy ball milling and to the uniform distribution of hard CNTs in the Al-alloy matrix. The hardness values of the composite as well as the matrix alloy compares well with that predicted by the Hall-Petch relationship.

Chapter-5 deals with powders of as-cast ingots of Al–25 at.%Fe and Al-34.5 at.%Fe alloys close to Al_3Fe and Al_2Fe intermetallic phases subjected to high energy ball milling to understand the possibility of formation of amorphous and/or nanocrystalline phases or any other metastable phases. The development of microstructure, the evolution of the various metastable phases and their stability are investigated by x-ray diffraction (XRD), differential scanning calorimetry (DSC) and transmission electron microscopy (TEM) techniques. Mechanical milling of the alloys up to 50 h was carried out in high energy planetary ball mill. It resulted in phase transformation from monoclinic and triclinic structures of Al_3Fe and Al_2Fe, respectively, to orthorhombic structure pertaining to Al_5Fe_2 phase and structural transformation from crystalline to amorphous phase. Hardness measurements revealed a transition from hardening to softening behavior in these mechanically milled alloys. The softening effect observed can be attributed to grain boundary sliding of the nano size grains as well as the evolution of amorphous phases.

Chapter-6 presents Al_5Fe_2 intermetallic phase processed by arc melting subjected to high energy ball milling to synthesize nanocrystalline and/or amorphous alloy. The milled powders were characterized using x-ray diffraction and transmission electron microscopy to study the sequential microstructural evolution and morphological variation. Hardness measurements were carried out on the individual powder particles to observe variation in mechanical properties with milling time. Microhardness shows an increase with grain size and reaches a maximum value of 9 Gpa at 20 h of milling, as expected from Hall-Petch (HP) relationship at least down to a grain size of 32 nm and then decreased with further refinement. The decrease in hardness could be an indication of softening, demonstrating the inverse Hall-Petch (IHP) behavior. Possible factors and deformation mechanism leading to softening behavior are discussed.

Chapter-7 presents a summary of the work indicating important finding arising out from the present investigation along with the suggestions for future work.