Synopsis

The aim of the present work is to engineer a new kind of coating material with superior quality and reliability in a cost effective way by laser cladding and ball milling process. Non equilibrium processing methods were adopted to produce different combination of phases, which are complicated, or cannot be produced by traditional fabrication processes. Advanced characterization techniques were utilized, to understand the mechanism of phase formation and the science behind our observations, thereby providing effective inputs for enhancement of outcomes.

The present work deals with the alloy systems of Fe-Sn at Fe-rich end (Fe-10 wt.% Sn) and Al-Cu-Fe-B quasicrystal composition ($Al_{61}Cu_{25}Fe_{12}B_2$) for laser cladding experiments and the Ni-Al system (corresponding to NiAl and Ni_3Al) for cladding by ball milling experiments. Fe-Sn intermetallics have very good hardness and magnetic properties; these properties were motivating factor to initiate the study to produce hard coatings. Among all crystalline and quasicrystalline phases of Al-Cu-Fe system, the λ and μ phases exhibit high hardness and are extremely brittle and are related to quasicrystals; the introduction of these phases into the Al matrix facilitated the production of the hard coatings of crystalline quasi-approximant phases on Al substrate. The presence of the intermetallic phases makes these coatings hard, and are good candidates for engineering applications. Since many years, Nickel aluminides were used as materials for high temperature and structural applications. These intermetallics also exhibit good thermal and oxidation properties and have good wear resistance. These properties of the intermetallics make them good candidates for coating applications. We have synthesized intermetallic particles embedded in different coating matrix. We have synthesized the coating of these composite materials which results in hard coatings with excellent strength.

The thesis is subdivided into five chapters. After introducing the thesis, the second chapter contains a review of literature. It gives a brief introduction to the present problems in research. The literature review presents the current synthesis methods of thick coatings, the emerging trends in new coating methods and a brief discussion on the recent developments in coating materials. More emphasis was given to the laser prototyping, the thermal barrier coatings and the functional graded coatings.

The next chapter describes the experimental techniques, the processing parameters and the characterization tools, used for understanding the microstructure evolution. This includes a brief descriptions of the alloys studied. A brief discussion of the techniques for the characterization of structure and microstructure is included in this chapter. I have also included in this chapter the challenges faced during sample preparations for different characterization techniques.

The fourth chapter discusses the formation of Fe-Sn intermetallic dispersions in iron matrix by laser cladding and remelting experiments. The evolution of the secondary phase particles suggest that liquid phase separation is a major contributor. The choice of Fe-Sn alloys as clad is dictated by the fact that Fe-Sn phase diagram exhibits a miscibility gap at high temperature. Sn forms a monotectic system with iron at a composition of Fe-34 at.% Sn and a temperature of 1130 °C followed by a two phase regions of tin rich and tin lean liquid. There is a large region where liquid tin coexists with α-Fe. As the two phase alloy is cooled, the system depending on the composition undergoes a series of peritectic reactions yielding the intermetallic phases of iron and tin. We have chosen a composition of Fe 10 wt.% Sn (Fe-5 at.% Sn) for our cladding experiments. Several clad and remelt samples were prepared to understand the effect of the cooling rate on phase formations. Majority of the intermetallic phases observed in the coatings are Fe_3Sn and

$FeSn_2$, while a large amount of Fe_3Sn phase was observed in the remelt layer. Re-solidification through laser surface processing, provides an opportunity to obtain quantitative information about the solidification process and for testing the prevalent theories of phase evolution. Growth velocities of the solidification front were calculated for the clad and remelt tracks. Several clad and remelt layers are analyzed by the EBSD technique, the results of which do not suggest any significant texture during the solidification process. The composition analysis of the dispersed particles was carried out by the EDS and EPMA. Sn is primarily segregated in the cell boundaries and near the cell boundaries in the dispersed particles. With increasing laser scan rate, we observe more regions containing dispersed particles with a composition close to that of Fe_3Sn. The dispersed phases are primarily Fe_3Sn; however an elongated morphology at the cell boundary indicates a mixture of phases. The TEM studies on a very large number of dispersed particles confirm that most of them are Fe_3Sn, and occasionally FeSn and $FeSn_2$.

In the fifth chapter we have described our attempt to synthesize a quasi-crystalline and approximant phases based on Al-Cu-Fe system. We have added a small amount of boron, to evaluate its effect on the resulting microstructure and phase formation. The quasi crystals lack ductility, which poses a major challenge in the processing. It was felt that the surface alloying techniques, in particular, laser cladding and remelting techniques can overcome this difficulty and can be adopted to utilize the properties of quasicrystals and its approximants. Most of the work that we have reported in this thesis deals with the Al rich end of the QC alloy. We have studied the effect of dilution from the Al substrate on the phase formation. Annealing experiments were carried out to study the effect of heat treatment on the phase formation

The alloy system of $Al_{61}Cu_{25}Fe_{12}B_2$ was taken for the current laser cladding experiments. The coatings were prepared by using the two step cladding process. In the first step, coatings were prepared at different laser power and clad speeds. In the second step some of the coatings were remelted at different traverse speeds to achieve different growth conditions. A few multi track samples were also processed at different processing conditions to understand the microstructural evolution. Tribological studies were carried out on some of the multi track clad and remelt samples to understand the wear behavior of coatings. Micro hardness measurements were done along the cross section of the samples, to evaluate the mechanical properties of the coatings.

The sixth chapter contains two major parts. The first part deals with the synthesis and characterization of Ni-Al intermetallic particles by ball milling process. Comparative studies of the ball milled powders were carried out using high energy ball mills (P7 & SPEX and P5). Rietveld whole powder pattern fitting was carried out on powder particles obtained at different intervals of time. Typical parameters like crystallite size, lattice parameter, phase fraction and strain were calculated by this method. Further studies on milled powders were carried out by transmission electron microscope to understand the mechanism of phase formation. We show that intermetallic compound NiAl and Ni_3Al can be synthesized using P7 and SPEX mills. However, the phases do not form in P5 mills under the conditions adopted by us. The second part of this chapter contains the preparation of Ni-Al intermetallic coatings using previously milled powders by P7, SPEX and P5 mills as well as fresh Ni and Al elemental powders. Comparative studies were carried out on coatings prepared by dry milling and wet milling method as well as between coatings prepared by the previously milled powders and the elemental mixture of Ni and Al powders. Coatings cross sections were examined to understand the microstructure within the coatings. As the annealing increases the diffusion of species, annealing experiments was conducted on coatings to produce better intermetallic coatings.